

FINAL REPORT

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**IOWA DEPARTMENT OF TRANSPORTATION
DES MOINES, IOWA**

**STRESS CORROSION TESTING OF SA 455 CARBON STEEL IN
METH-REACTION INHIBITED AGRICULTURAL AMMONIA SOLUTIONS**

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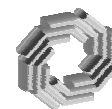
STRESS CORROSION TESTING OF SA 455 CARBON STEEL IN METH-REACTION INHIBITED AGRICULTURAL AMMONIA SOLUTIONS

PREPARED FOR
IOWA DEPARTMENT OF TRANSPORTATION
DES MOINES, IOWA

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This report documents work performed by CC Technologies, Inc. (*CC Technologies*) Dublin, Ohio, for the Iowa Department of Transportation – Motor Vehicle Division (*Iowa DOT*) and the Iowa Department of Agriculture and Land Stewardship (IDALS), Des Moines, IA, under the terms of Contract Agreement Number 0901MVE (Project # MH05-19-1, DUNS # 12-052-7275).

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*Stress Corrosion Testing of SA 455 Carbon Steel
in Meth-Reaction Inhibited Agricultural Ammonia Solutions*

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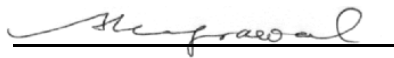
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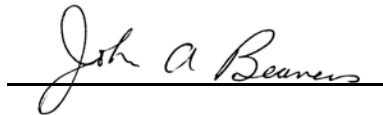
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EXECUTIVE SUMMARY

Agricultural ammonia is a compound used in the illegal manufacture of methamphetamine (meth). The Iowa Department of Transportation – Motor Vehicle Division (Iowa DOT) and the Iowa Department of Agriculture and Land Stewardship (IDALS) - Fertilizer Division, Des Moines, IA are working with the Iowa Anti-Meth Task Force and the Agribusiness Association of Iowa (AAI) to develop meth-reaction inhibiting compounds (additives) that can be added to agricultural grade anhydrous ammonia.

Iowa State University showed that an addition of 2% calcium nitrate ($\text{Ca}(\text{NO}_3)_2$) can effectively inhibit the meth-reaction in liquid anhydrous ammonia. However, addition of a meth-inhibiting compound should not compromise the structural integrity of the base metal and welds of carbon steel tanks that are used to store and transport anhydrous ammonia.

Preliminary testing showed that general corrosion of carbon steel in ammonia containing 2% calcium nitrate was minimal, but it was not known if this environment increases susceptibility to stress corrosion cracking (SCC). It was also unknown if an increase in SCC susceptibility occurs if nitrogen stabilizer N-Serve® is also present in ammonia. Cracking in ammonia nurse tanks, especially at non-post weld heat treated (non-PWHT) welds, has received increased attention since the catastrophic failure of a nurse tank long-seam weld in Calamus, Iowa in April 2003.

SA 455 carbon steel is typically used for the construction of nurse tanks, with nurse tank heads being stress relieved and the long seam welds not stress relieved. SA 455 carbon steel contains approximately 0.33% carbon (C), 1% manganese (Mn) and no other alloying additions. Other steels used in the storage and transportation of agricultural grade ammonia include SA 516 Gr. 70 steel, typically used for storage tanks with all welds stress relieved. SA 517 Gr. B or Gr. E steel is typically used for transport trailers and is a quenched and tempered material. SA 612 steel is typically used for rail cars. The present work was focused on the nurse tank steel SA455.

In this project, IDALS requested CC Technologies, Inc. (*CC Technologies*), Dublin, Ohio to perform stress corrosion tests under the terms of Contract Agreement Number 0901MVE (Project # MH05-19-1, DUNS # 12-052-7275).

The objectives of the work were to evaluate the effects of 2% $\text{Ca}(\text{NO}_3)_2$, N-Serve®, and welding on stress corrosion cracking (SCC) susceptibility of SA 455 carbon steel in agricultural anhydrous ammonia solutions containing 0.2% water.

The scope of the work consisted of a total of eighteen (18) mechanical tests in anhydrous ammonia solutions. Duplicate tests were performed on specimens machined from three portions of a welded SA455 carbon steel plate: base metal, weld fusion metal, and heat affected zone (HAZ). Three different anhydrous ammonia solutions were used to test the specimens. The mechanical properties of each specimen were measured, and visual and metallographic examination was performed to inspect for the presence of cracks.

Key test results and conclusions, based on the results, are presented below:

1. Ammonia SCC was found on SA 455 carbon steel specimens in all three environments investigated: (1) anhydrous ammonia containing 0.2% water, (2) with added 2% calcium nitrate, and (3) with added 2% calcium nitrate and N-Serve®.
 - a. The severity (depth) of SCC did not appear to be related to the type of environment used in the tests.
 - b. Two percent calcium nitrate additive did not increase SCC susceptibility.
2. Ammonia SCC was observed in the base metal of four specimens – three of which were base metal specimens, and one was a HAZ specimen.
 - a. The weld fusion metal and HAZ did not have increased SCC susceptibility, as compared with the base metal.
 - b. The SCC appeared as relatively small fissures on the specimen surfaces.
 - c. The depth of the cracks ranged from 50-1100 μm .
3. Crack severity was more affected by the metal microstructure than by the variations in environment composition.
4. The deepest cracks were preferentially located in a hard phase (362 HK), which was different from the base metal matrix of ferrite and pearlite. The phase etched brown with Nital and likely was martensite. The base metal microstructure of ferrite/pearlite experienced no SCC in the same specimens.

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APPENDIX

Appendix A – Procedure for SCC Tests in Anhydrous Ammonia

LIST OF ABBREVIATIONS

CS	carbon steel
DSAW	double submerged arc weld
HAZ	heat affected zone
HK	Knoop hardness
ID	inside surface
LVDT	linear variable displacement transducer
OD	outside surface
OSHA	occupational safety and health administration
PEL	permissible exposure limit
PWHT	post-weld heat treatment
SCC	stress corrosion cracking
SEM	scanning electron microscope
SSR	slow strain rate
STEL	short term exposure limit
TWA	time weighted average
UTS	ultimate tensile strength
YS	yield strength

1.0 – INTRODUCTION

This report documents work performed by CC Technologies, Inc. (CC Technologies), Dublin, OH, for the Iowa Department of Transportation – Motor Vehicle Division (Iowa DOT) and the Iowa Department of Agriculture and Land Stewardship (IDALS) - Fertilizer Division, Des Moines, IA, under the terms of Contract Agreement Number 0901MVE (Project # MH05-19-1, DUNS # 12-052-7275).

Agricultural ammonia is a compound used in the illegal manufacture of methamphetamine (meth). Iowa DOT and IDALS are working with the Iowa Anti-Meth Task Force and the Agribusiness Association of Iowa (AAI) to develop meth-reaction inhibiting compounds (additives) that can be added to agricultural grade anhydrous ammonia. However, addition of a meth-inhibiting compound should not compromise the structural integrity of the base metal and welds of carbon steel tanks that are used to store and transport anhydrous ammonia.

Research conducted at Iowa State University resulted in the compilation of a list of five possible meth-inhibiting additives, and their general corrosion properties were tested by exposing steel coupons to ammonia containing 2% additive for 30 days. It was shown that 2% calcium nitrate ($\text{Ca}(\text{NO}_3)_2$) in liquid ammonia was an effective meth-inhibitor that caused little general corrosion of carbon steel. Calcium nitrate is relatively inexpensive and a readily available chemical.

Although general corrosion in calcium nitrate containing ammonia was minimal, it was not known if this environment increases susceptibility of carbon steel to stress corrosion cracking (SCC). Cracking in ammonia nurse tanks, especially at non-post weld heat treated (non-PWHT) welds, has received increased attention since the catastrophic failure of a nurse tank long-seam weld in Calamus, Iowa in April 2003 [Ref. 1]. Therefore, a number of ammonia nursing tanks has been treated with suspended calcium nitrate since 2002. Two tanks started to be treated and monitored for corrosion in the fall of 2002 (one is still being treated) and nine more tanks have been treated since the summer of 2003.

Another compound often added to agricultural ammonia is nitrogen stabilizer N-Serve®,⁽¹⁾ which controls the rate of de-nitrification and leaching to soil. It was unknown if the presence of N-Serve® in anhydrous ammonia increases SCC susceptibility when 2% $\text{Ca}(\text{NO}_3)_2$ is also present.

SA 455 carbon steel is typically used for the construction of nurse tanks, with nurse tank heads being stress relieved and the long seam welds not stress relieved. SA 455 carbon steel contains approximately 0.33% carbon (C), 1% manganese (Mn) and no

¹ N-Serve® is a registered trademark of Dow AgroSciences.

other alloying additions. Other steels used in the storage and transportation of agricultural grade ammonia include SA 516 Gr. 70 steel, typically used for storage tanks with all welds stress relieved. SA 517 Gr. B or Gr. E steel is typically used for transport trailers and is a quenched and tempered material. SA 612 steel is typically used for rail cars. The present work was focused on the nurse tank steel SA455.

SCC in carbon steel (CS) exposed to anhydrous ammonia is a known phenomenon [Ref. 2] and aspects such as solution chemistry, steel composition, and the influence of air and water have been researched and documented [Ref. 3]. Crack growth rates for base metal and welds were investigated in another study [Ref. 4]. It was found that residual stresses around welds increase cracking susceptibility, while microstructure variations in the weld and heat affected zone (HAZ) seemed to have little effect. In addition, cracking has been observed in storage vessels in the vapor space above water doped ammonia. While pure ammonia does not cause SCC, contamination with air is the primary cause of SCC. Cracking susceptibility appears to increase with the strength of the steel. The addition of minimum 0.1 weight percent of water to ammonia inhibits cracking in the liquid phase. Standard practice to avoid SCC in carbon steel exposed to anhydrous ammonia includes additions of 0.2% water by weight [Ref. 5]. In the present work, 0.2% water was used in the anhydrous ammonia solutions.

2.0 – OBJECTIVES AND SCOPE

The objectives of the work were to evaluate the effects of 2% $\text{Ca}(\text{NO}_3)_2$, N-Serve®, and welding on stress corrosion cracking (SCC) susceptibility of SA 455 carbon steel in agricultural anhydrous ammonia solutions containing 0.2% water.

The scope of the work consisted of a total of eighteen (18) mechanical tests in anhydrous ammonia. Duplicate tests were performed on SA455 carbon steel (CS) base metal, weld fusion metal, and heat affected zone (HAZ), in three different anhydrous ammonia solutions. After the tests, the specimens were examined metallographically for the presence of cracks.

3.0 – APPROACH

Material for Testing

A 24-inch × 24-inch × 0.341-inch thick sample plate of welded SA 455 material was provided by a nurse tank manufacturer,⁽²⁾ see Figure 1. It was reported that the plate was fabricated using the same procedures that would be applied for welds in nurse tank manufacturing. The weld in the plate was not subjected to PWHT. It was also reported that, prior to shipping to CC Technologies, the weld was inspected by full radiography

² Trinity Industries, Inc., 2525 Stemmons Freeway, Dallas, TX 75207-2401, Tel. 214-631-4420.

under supervision of the tank manufacturer to confirm that no unacceptable defects were present.

A metallographic cross-section through the weld was prepared to document its appearance and microstructure in the as-received condition. The cross-section was prepared using standard metallographic procedures, consisting of increasingly fine grinding steps on SiC abrasive paper, with a final polish of 3 μm diamond paste. A Nital etchant was used to reveal the microstructure. The weld was examined at 25X magnification using a Nikon SMZ800 Zoom Stereomicroscope.

Test Environments

Iowa DOT supplied three different ammonia solutions for the tests in standard commercial gas cylinders of approximately two gallons internal volume. It was reported that each cylinder was prepared with agricultural grade ammonia containing 0.2% water by weight. The concentration of N-Serve® was not given. The environments for the tests were (1) agricultural grade anhydrous ammonia without additions, (2) anhydrous ammonia with addition of 2% calcium nitrate, and (3) ammonia with addition of both 2% calcium nitrate and N-Serve®, as listed in Table 1.

Test Specimens

The specimens were machined from the representative welded plate, to closely replicate actual welds in agricultural ammonia tanks. Round-bar tensile test specimens were machined from the plate at locations relative to the weld as shown in Figure 2. A typical specimen is shown in Figure 3. The overall dimensions of the specimens were 2.5-inch long, 0.250-inch diameter, with a gage section of 1-inch long, 0.125-inch diameter, see Figure 4. One set of eight specimens was all-base metal, Figure 4a, a second set of eight specimens had the approximate middle of the weld HAZ at the center of the gage length, Figure 4b, and the third set of eight specimens had the weld fusion line at the center of its gage length, Figure 4c.

The test matrix consisted of a total of 18 tests, as shown in Table 2. Two base metal, two HAZ specimens, and two weld metal specimens were tested in each of the three environments. Twenty-four (24) test specimens were prepared from the SA 455 steel plate.⁽³⁾ Eighteen (18) specimens were used for testing, one specimen was used for a trial run, and five (5) specimens remained unused.

Test Set-Up

SCC testing was conducted using the slow strain rate (SSR) technique, following procedures similar to those described in Standard Practice ASTM G129 [Ref. 6]. In the tests, the tensile specimen was immersed in the test environment and strained to failure

³ Metal Samples, P.O. Box 8, 152 Metal Samples Road, Munford, AL 36268, Tel. 256-358-4202.

at a slow rate. The time-to-fracture, tensile curve (plot of strain vs. stress) and ultimate tensile strength were measured for each test. The percent elongation, reduction in area, and the deepest surface fissure in the specimen were measured after the test.

A diagram of the test cell is shown in Figure 5. The specimens had a machined surface rather than “as welded.” To simulate the presence of corrosion products on the tank’s surface, a pre-corroded coil spring was placed around each machined specimen. The carbon steel specimen with the spring was then threaded into carbon steel pull rods that inserted in a stainless autoclave through Teflon sliding pressure seals. The pull rods were connected to the SSR machine to apply the tensile load. The pull rod assembly with the specimen and spring was electrically isolated from the test cell. Ports for filling and emptying the autoclave and a pressure gage and pressure relief valve were incorporated in the autoclave.

To simulate conditions of agricultural ammonia storage, the test cell was purged with argon to exclude oxygen. The autoclave was then filled with ammonia solution to a predetermined level, as verified by a sight glass mounted on the autoclave. The fill height was such that the entire gage length of the specimen was immersed in the ammonia solution. To verify this, the location of the test cell, (Figure 6a) was compared with the location of the specimen in the pull rods, (Figure 6b). After each test, the used quantity of anhydrous ammonia was disposed by diluting and neutralizing it.

Figure 7 shows an overall view of the test setup. The SSR machine, the ammonia cylinder and the disposal carboy were all located inside a chemical fume hood. The computer controlling the machine and the cylinder with argon were located beside it. In each test, approximately 800 ml of solution was used. All the tests were conducted at a constant extension rate of 1.0×10^{-6} in./sec., at room temperature and at equilibrium pressure of ammonia (approximately 110 psig). A detailed test procedure is provided in Appendix A.

Post-Test Analysis

Following testing, the specimens were visually inspected for the presence of cracks, using a Nikon SMZ800 Zoom Stereomicroscope with magnifications up to 30X and photographed. If no cracks or other surface features (possible incipient cracks) were visible with this procedure, then one-half of the specimen gage section was longitudinally sectioned, and the section was mounted in epoxy resin. These mounts were prepared using standard metallographic procedures, consisting of increasingly fine grinding steps on SiC abrasive paper, with a final polish of 1 μ m diamond paste.

Specimens with observable surface features (fissures) were marked and then prepared similarly by polishing half-way through the observed surface feature. The prepared cross-sections were examined using a Nikon Epiphot-TME Inverted

Metallurgical Microscope with magnifications between 25X and 400X. The specimens were examined in the as-polished condition and in the etched condition to determine where the crack(s) was located relative to the grain structure and weld regions. The results were documented in photomicrographs.

Safety Considerations

Handling of anhydrous ammonia for testing, test cell loading and unloading, and disposal required special safety procedures. A protective suit, goggles, and face shield were utilized by the technician. A self-contained breathing apparatus was also available for exposure situations. CC Technologies' laboratory has ventilated hoods for testing hazardous and corrosive chemicals. The mechanical testing machine was placed under a hood for the SSR tests. A line-AC powered ammonia detector with battery backup was installed to trigger at the OSHA PEL⁽⁴⁾ [time weighted average (TWA) = 50 ppm (35 mg/m³)] and OSHA STEL⁽⁵⁾ [TWA = 35 ppm (24 mg/m³)]. After each test, the used quantity of anhydrous ammonia was disposed by diluting and neutralizing it.

4.0 – RESULTS AND DISCUSSION

Pre-Test Microstructures

Figure 8 shows an optical photomicrograph of a metallographically prepared cross-section through the weld. The weld is a double submerged arc weld (DSAW), made with two weld beads – one from either side of the plate.

Examples of the microstructures present at different locations near and in the weld are shown in Figure 9. These photomicrographs were taken from Specimen 2. The plate's base metal (a) had a banded microstructure of ferrite (light constituent) and pearlite (dark constituent). Banding results from manufacturing the steel plate when it is hot-rolled to its final shape. Qualitatively, the amount of pearlite appears consistent with approximately 0.3% C in the carbon steel. The HAZ near the base metal (b) also reveals the banded structure, and ferrite and pearlite are visible. However, the grain size is much finer in this area of the HAZ than in the base metal, indicating that normalizing has taken place. The center of the HAZ (c) contains larger grains, and the ferrite and pearlite are not banded. This microstructure is consistent with grain growth following normalizing (austenitizing) as a result of heating. The HAZ near the weld (d) showed large pearlite grains with grain boundary ferrite along prior austenite grain boundaries and Widmanstätten ferrite (plate-like) within the pearlite grains. Again, this is a microstructure caused by heating. The weld fusion metal near the HAZ (e) and the weld fusion metal at the weld center (f) both have a solidification microstructure with fine pearlite (dark) in a matrix of ferrite (light). The grains are elongated along the direction of maximum heat flux, which occurred during weld solidification.

⁴ PEL = permissible exposure limit

⁵ STEL = short term exposure limit

Tensile Properties

The results of the SSR tests organized by each of the three environments are listed in Table 3, and the same data are listed once more organized by material in Table 4. Six specimens were tested for each condition: 2 base metal, 2 HAZ and 2 weld fusion metal. The tables give the time-to-fracture measured during the test, the ultimate tensile strength as calculated from the recorded load-displacement data, percent elongation and reduction in area as measured from the specimens after the tests, and the size of the deepest fissure found in a longitudinal cross-section from each specimen.

The tensile requirements for SA 455 steel [Ref. 7] for material up to 0.375 inch thickness are 38 ksi minimum yield strength (YS), 75-95 ksi ultimate tensile strength (UTS), 22% elongation in two-inch long sections, and 15% elongation in eight-inch long sections.

The ranges of measured tensile properties are listed in Table 5 and the associated stress-strain curves are shown in Figures 10a – 10c. For the base metal, the UTS (96.6 – 102.2 ksi) exceeded the specification, and the elongation (19.0 – 21.0%) was slightly lower as compared with the specification. These variations are small and not significant to assess SCC susceptibility of the specimens.

The mechanical properties for the SSR tests did not provide a clear indication whether or not SCC had occurred. There were differences among the specimens based on time-to-fracture and elongation that correlated with the metal microstructure (base metal, HAZ, weld metal) but not test environment. Base metal specimens had the longest time-to-fracture and largest total elongation. HAZ specimens had the shortest time-to-fracture and the smallest total elongation. Weld metal specimens had times-to-fracture and total elongations in between the base metal and HAZ specimens. While some variation is expected, even for identical specimens made from the same material, a specimen experiencing significant SCC usually fractures in an appreciably shorter time. The time-to-fracture for all tests showing SCC were similar to the other tests in the present work. Thus, visual inspection and examination of metallographic cross-sections was necessary to confirm the presence or absence of cracks.

Visual Inspection

Figures 11, 12, and 13 show photographs of the gage sections of the fractured specimens alongside photomicrographs of the longitudinal cross-sections of the respective fracture tips. All specimens failed in the gage section, and all had significant necking to form cup and cone fracture appearance, which is indicative of overall ductile failure. With the exception of the shiny metallic specimens from Test 15 (base metal – ammonia) and Test 14 and 17 (base metal and weld metal in ammonia + 2% $\text{Ca}(\text{NO}_3)_2$), the specimens tested in anhydrous ammonia and anhydrous ammonia + 2% calcium

nitrate had a gray/black surface scale and scattered orange corrosion spots. The spots appeared to have formed as flash-rust immediately after removing the specimens from the test cell. All specimens tested in solutions containing N-Serve® retained a metallic luster after the test.

Metallographic Examination

At higher magnifications, small surface fissures were revealed on the specimens from Tests 14, 17, and 12, as shown in Figures 14, 15, and 16, respectively. For these specimens, the longitudinal cross-sections were prepared by cutting through these features. For all other specimens, no notable surface features were visible and a random longitudinal cross-section was made halfway through the gage thickness.

Figures 17a-c present photomicrographs of the deepest surface fissures in the cross-sections of the various gage sections. The measurement of the maximum penetration depth is also shown in these photomicrographs. The specimens from Tests 17, 5, 14, and 12 likely experienced SCC. The cracks were present in 3 base metal specimens (anhydrous ammonia and anhydrous ammonia + $\text{Ca}(\text{NO}_3)_2$) and in 1 HAZ specimen (anhydrous ammonia + $\text{Ca}(\text{NO}_3)_2$ + N-Serve®). The cracks encompassed multiple grains and ranged in depth between 50-1100 μm . The etched microstructure at the cracks showed that the cracks were intergranular in nature, consistent with ammonia SCC. All other penetrations were possibly incipient cracks. The incipient cracks (ten specimens) generally encompassed two grains or less, and had depth < 50 μm . No significant surface features were found in the remaining four specimens.

Figures 18, 19, and 20 show additional photomicrographs of cross-sections for the specimens with the deepest cracks (Tests 14, 17, and 12). It was noticed that, in addition to the ferrite and pearlite in the banded microstructure, another phase was present at the locations where cracks were present. This phase appeared light-brown after Nital etching, while the ferrite was light (white) and the pearlite dark (black). Knoop micro-hardness (HK) measurements were taken to distinguish it from its surroundings. Indeed, the hardness of the brown phase was 362 HK, while the pearlite gave readings in the range of 180-290 HK, and the ferrite was 220-270 HK. Thus, the distinct phase may have been martensite – a hard phase that may be present as a remnant of the steel plate's manufacturing process. The cracking appeared to have occurred preferentially within this phase, as multiple parallel cracks were confined inside the distinct phase in each of these three specimens. The base metal microstructure of ferrite/pearlite was found not to be affected by SCC in these same specimens.

Figures 21a – Figures 21c present photomicrographs showing the microstructure around the fracture area, to identify the location of the fracture relative to the weld and HAZ in each specimen. The figures demonstrate that the fracture location for each specimen was in the base metal, except Test 2. Test 2 was conducted on a weld metal

specimen in anhydrous ammonia, and the specimen failed at the weld center. The fact that all but one specimen failed in the base metal showed that under the test conditions, the weld fusion metal and HAZ did not have increased SCC susceptibility, as compared with the base metal.

SCC was found in all three test environments and the severity (depth) of cracking appears not to be related to the type of environment. All specimens fractured in the base metal, except for one weld specimen that fractured in the weld center. SCC was found in the base metal of four specimens – three of which were base metal specimens and was a HAZ specimen. Surface fissures were noticed on the gage sections of ten specimens whereas; the remaining four specimens were unaffected.

5.0 – CONCLUSIONS

Key test results and conclusions based on the results are presented below:

1. Ammonia SCC was found on SA 455 carbon steel specimens in all three environments investigated in this project: (1) anhydrous ammonia containing 0.2% water, (2) with added 2% calcium nitrate, and (3) with added 2% calcium nitrate and N-Serve®.
 - a. The severity (depth) of SCC did not appear to be related to the type of environment used in the tests.
 - b. Two percent calcium nitrate additive did not increase SCC susceptibility.
2. Ammonia SCC was observed in the base metal of four specimens – three of which were base metal specimens, and one of which was a HAZ specimen.
 - a. The weld fusion metal and HAZ did not have increased SCC susceptibility, as compared with the base metal.
 - b. The SCC appeared as relatively small fissures on the specimen surfaces.
 - c. The depth of the cracks ranged from 50-1100 μm .
3. Crack severity was more affected by the metal microstructure than by the variations in environment composition.
4. The deepest cracks were preferentially located in a hard phase (362 HK), which was different from the base metal matrix of ferrite and pearlite. The phase etched brown with Nital and likely was martensite. The base metal microstructure of ferrite/pearlite experienced no SCC in the same specimens.

6.0 – FUTURE WORK

In order to understand and prevent possible future SCC-related incidents and to understand the effects of agricultural ammonia additives, the following future work is suggested:

1. Investigate the SCC behavior of other steels commonly used in the agricultural ammonia storage and transportation industry when exposed to the meth-inhibiting additive 2% $\text{Ca}(\text{NO}_3)_2$, N-Serve®, and 0.2% water. These steels include:
 - a. SA 516 Gr. 70 for storage tanks,
 - b. SA 517 Gr. B or Gr. E for transport trailers, and
 - c. SA 612 for rail cars.
2. Investigate the effect of temperature on the SCC susceptibility of the various steels. (The temperature was 81°F at the time of the April 2003 Calamus accident and tanks experience higher temperatures on hot summer days.)
3. Investigate test materials fully submerged in ammonia solutions and at the liquid/vapor interface.
4. Perform some SCC experiments at a slower strain rate (e.g., $5 \times 10^{-7} \text{ sec}^{-1}$), to investigate the effect of longer exposure time in the environments.

7.0 – REFERENCES

1. “Nurse Tank Failure with Release of Hazardous Materials near Calamus, Iowa, April 15, 2003,” National Transportation Safety Board, Washington, DC, HAZMAT Incident Report NTSB/HZM-04/01, PB 2004-917001, Notation 7564A, Adopted June 22, 2004.
2. Jones, D.A. Principles and Prevention of Corrosion, Second Edition, Prentice Hall, Upper Saddle River, NJ, pp. 259-260, 1996.
3. Liv Lunde, “Stress Corrosion Cracking of Different Steels in Ammonia – With Special Emphasis on Vapour Phase Cracking,” Institute for Energy Technology, Norway, IFE-Report No. IFE/KR/E-83/007, Symposium on Safety in Ammonia Plants and Related Facilities, American Institute of Chemical Engineers, Denver, CO, August 29-31, 1983.
4. Liv Lunde and Rolf Nyborg, “SCC of Carbon Steel in Ammonia – Crack Growth Rate Studies and Means to Prevent Cracking,” NACE, CORROSION/89, Conference Paper 98, New Orleans, April 17-21, 1989.
5. “Avoidance of Stress Corrosion Cracking (SCC) in Cargo Tanks, Reliquefaction Condensers, and Condensate Return Pipework, With Liquefied Ammonia Cargoes,” Information Paper No. 2, Society of International Gas Tanker & Terminal Operators, Ltd., (SIGTTO), November 1998.
6. ASTM G129-00, “Standard Practice for Slow Strain Rate Testing to Evaluate the Susceptibility of Metallic Materials to Environmentally Assisted Cracking,” ASTM International, Book of Standards, Volume 03.02.
7. ASTM A455M-03, “Standard Specifications for Pressure Vessel Plates, Carbon Steel, High-Strength Manganese,” ASTM International, Book of Standards, Volume 01.04.

Table 1. Test environments and ammonia cylinder numbers.

Environment	Test Solution				Ammonia Cylinder №
	Ammonia	Water	N-Serve®	Ca(NO ₃) ₂	
1	yes	0.2%	no	no	LCCG-SG00-62
2	yes	0.2%	no	2%	GF204909A
3	yes	0.2%	yes	2%	M8004 REE454

(*) Test 2 had 0.4% water.

Table 2. Test matrix for evaluating the SCC susceptibility of SA 455 carbon steel specimens in various anhydrous ammonia solutions.

Environment		SA 455 Base Metal	SA 455 Weld Metal (no PWHT)	SA 455 HAZ (no PWHT)
1	Anhydrous Ammonia	2 Tests	2 Tests	2 Tests
2	Anhydrous Ammonia with 2% Ca(NO ₃) ₂	2 Tests	2 Tests	2 Tests
3	Anhydrous Ammonia with 2% Ca(NO ₃) ₂ , and N-Serve®	2 Tests	2 Tests	2 Tests

Table 3. Results of slow strain rate tests in various anhydrous ammonia solutions, organized by the environment.

Test №	Environment	SA 455 Material	Time To Fracture *	Ultimate Tensile Strength	Elongation	Reduction In Area	Deepest Surface Fissure	Appearance
			hours	ksi	%	%	µm	
4	Anhydrous Ammonia	Base Metal	60.3	101.2	20.8	64.8	47	Incipient Crack
17		Base Metal	58.0	101.9	20.1	60.4	345	Crack
3		HAZ	48.7	103.0	16.7	61.8	20	Incipient Crack
15		HAZ	44.2	101.5	15.2	60.1	11	Incipient Crack
2		Weld Center	54.8	96.3	18.3	65.0	< 5	No Cracks
16		Weld Center	50.4	101.5	17.4	60.8	11	Incipient Crack
5	Anhydrous Ammonia with 2% Ca(NO ₃) ₂	Base Metal	60.6	99.9	21.7	62.4	52	Crack
14		Base Metal	56.3	97.6	19.0	53.0	138	Crack
6		HAZ	45.1	104.3	15.8	58.8	< 5	No Cracks
18		HAZ	42.5	92.9	14.5	65.0	8	Incipient Crack
7		Weld Center	50.3	103.0	17.7	62.8	23	Incipient Crack
19		Weld Center	51.8	96.8	17.6	63.4	20	Incipient Crack
8	Anhydrous Ammonia with 2% Ca(NO ₃) ₂ and N-Serve®	Base Metal	58.7	102.2	20.9	63.8	8	Incipient Crack
11		Base Metal	60.2	99.9	21.0	66.3	12	Incipient Crack
9		HAZ	45.1	102.0	15.5	61.8	< 5	No Cracks
12		HAZ	44.9	102.9	15.1	64.4	1097	Crack
10		Weld Center	54.0	100.7	19.3	64.4	< 5	No Cracks
13		Weld Center	46.4	97.2	15.4	58.4	29	Incipient Crack

* All tests were conducted at a constant extension rate of 10^{-6} sec^{-1} and at room temperature.

Table 4. Results of slow strain rate tests in various anhydrous ammonia solutions, organized by the specimen material.

SA 455 Material	Environment	Test No	Time To Fracture *	Ultimate Tensile Strength	Elongation	Reduction In Area	Deepest Surface Fissure	Appearance
			hours	ksi	%	%	µm	
Base Metal	Anhydrous Ammonia	4	60.3	101.2	20.8	64.8	47	Incipient Crack
		17	58.0	101.9	20.1	60.4	345	Crack
	Anhydrous Ammonia with Ca(NO ₃) ₂	5	60.6	99.9	21.7	62.4	52	Crack
		14	56.3	97.6	19.0	53.0	138	Crack
	Anhydrous Ammonia with Ca(NO ₃) ₂ and N-Serve®	8	58.7	102.2	20.9	63.8	8	Incipient Crack
		11	60.2	99.9	21.0	66.3	12	Incipient Crack
HAZ	Anhydrous Ammonia	3	48.7	103.0	16.7	61.8	20	Incipient Crack
		15	44.2	101.5	15.2	60.1	11	Incipient Crack
	Anhydrous Ammonia with 2% Ca(NO ₃) ₂	6	45.1	104.3	15.8	58.8	< 5	No Crack
		18	42.5	92.9	14.5	65.0	8	Incipient Crack
	Anhydrous Ammonia with 2% Ca(NO ₃) ₂ and N-Serve®	9	45.1	102.0	15.5	61.8	< 5	No Cracks
		12	44.9	102.9	15.1	64.4	1097	Crack
Weld Center	Anhydrous Ammonia	2	54.8	96.3	18.3	65.0	< 5	No Cracks
		16	50.4	101.5	17.4	60.8	11	Incipient Crack
	Anhydrous Ammonia with 2% Ca(NO ₃) ₂	7	50.3	103.0	17.7	62.8	23	Incipient Crack
		19	51.8	96.8	17.6	63.4	20	Incipient Crack
	Anhydrous Ammonia with 2% Ca(NO ₃) ₂ and N-Serve®	10	54.0	100.7	19.3	64.4	< 5	No Cracks
		13	46.4	97.2	15.4	58.4	29	Incipient Crack

* All tests were conducted at a constant extension rate of 10^{-6} sec^{-1} and at room temperature.

Table 5. Range of tensile properties obtained in the SSR tests compared to SA 455 steel specifications.

Parameter	Base Metal	HAZ	Weld Metal	SA 455 Steel Specification (up to 0.375-in. thickness)
Time-to-fracture, hours	58.0 – 60.6	42.5 – 48.7	46.4 – 54.8	not applicable
Ultimate tensile strength, ksi	96.6 – 102.2	92.9 – 104.3	96.3 – 103.0	75 – 95
Elongation, %	19.0 – 21.0	14.5 – 16.7	15.4 – 19.3	Min. 22% (in 2-inches)
Reduction in Area, %	53.0 – 66.3	58.8 – 65.0	58.4 – 65.0	not specified



Figure 1. Welded SA 455 plate before specimen manufacture.

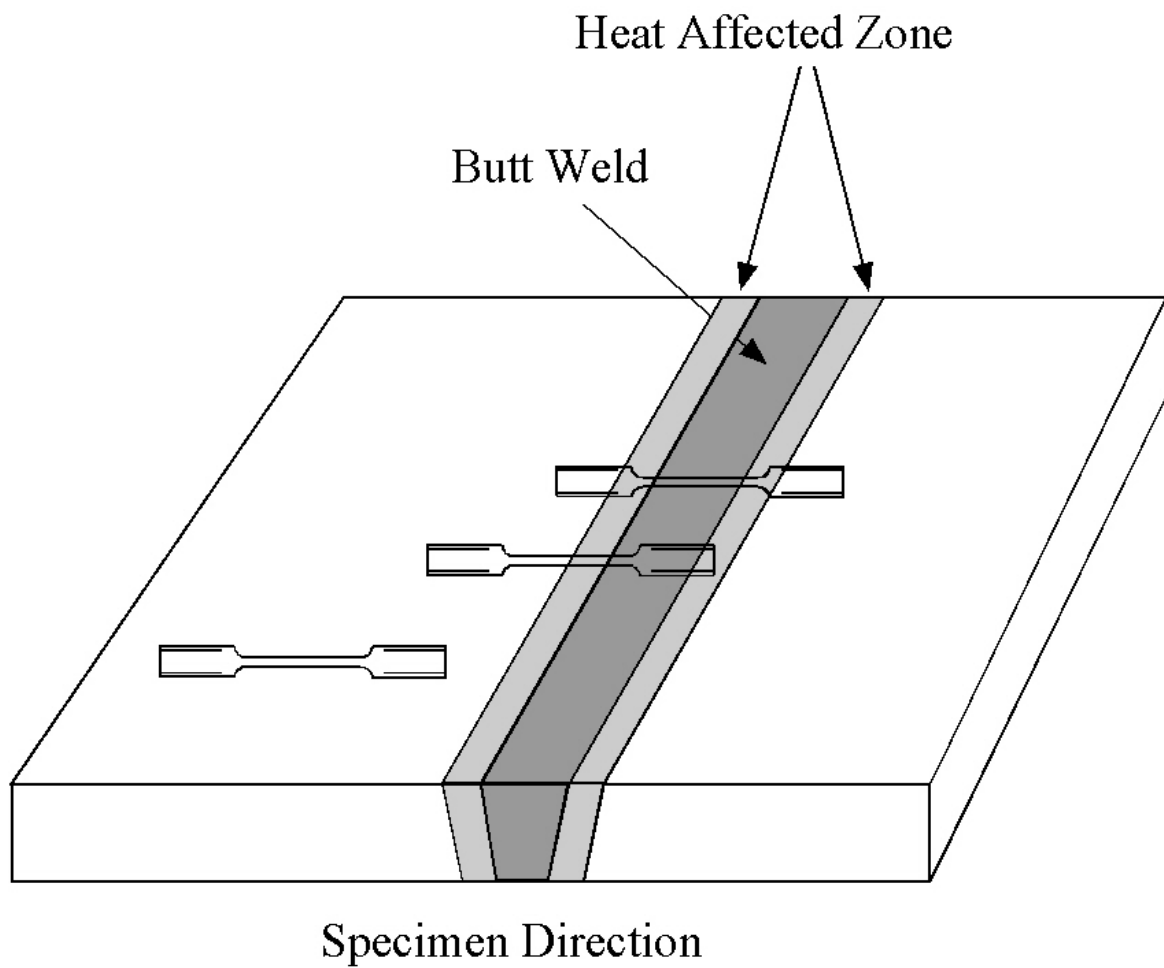


Figure 2. Sketch of welded plate for specimen fabrication.

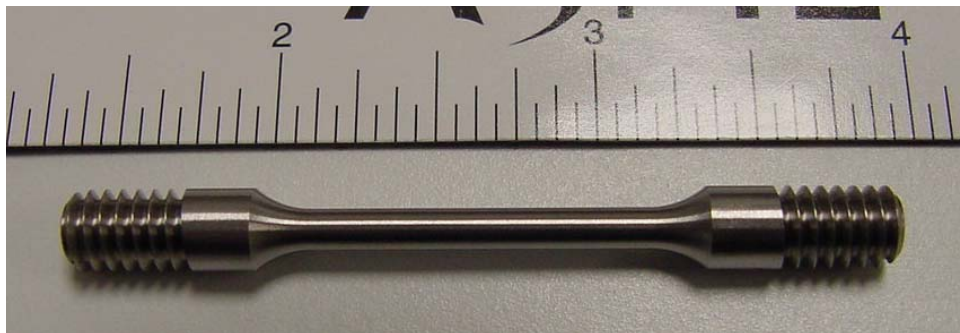
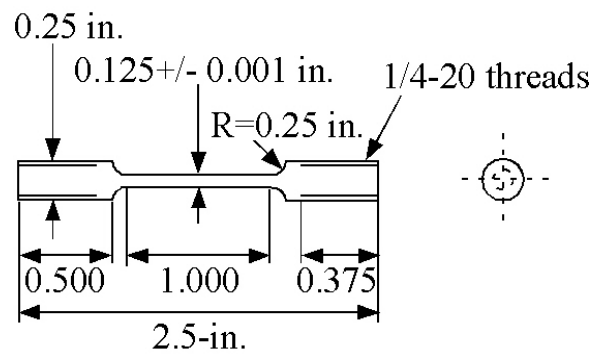
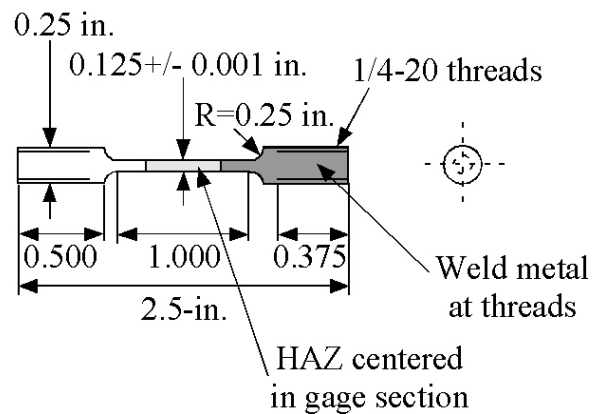


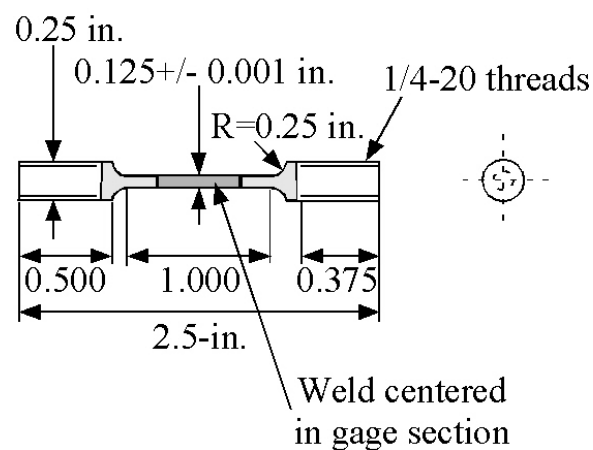
Figure 3. Typical test specimen.



a) Base metal specimen.



b) HAZ specimen.



c) Weld metal specimen.

Figure 4. Drawings showing three difference types of specimens incorporating the base metal, HAZ, and weld.

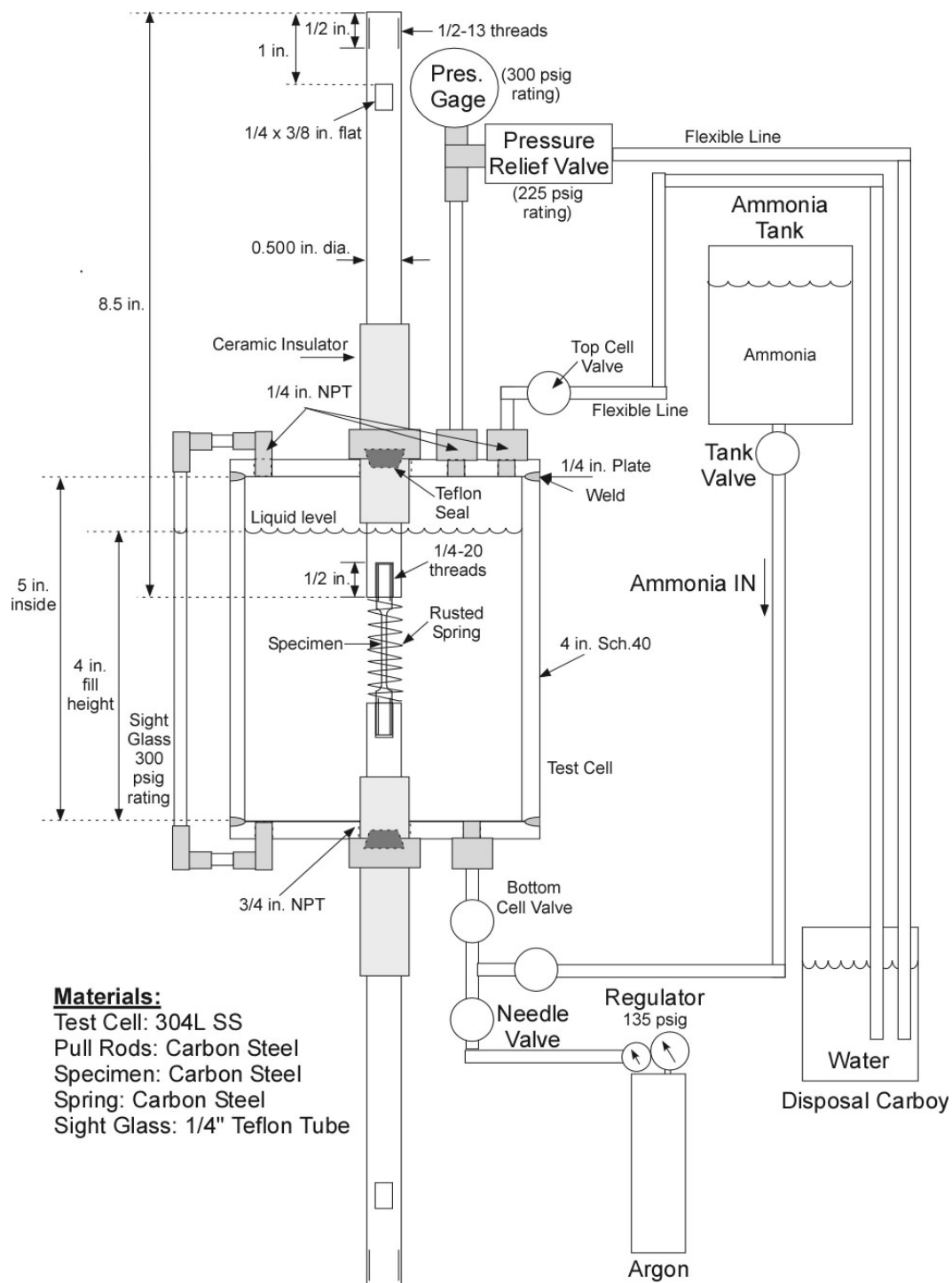


Figure 5. Diagram of test cell.



Figure 6. Photographs of cell assembly and a specimen shown positioned in SSR machine, but without the cell.



Figure 7. Test setup in SSR machine and associated computer control system.

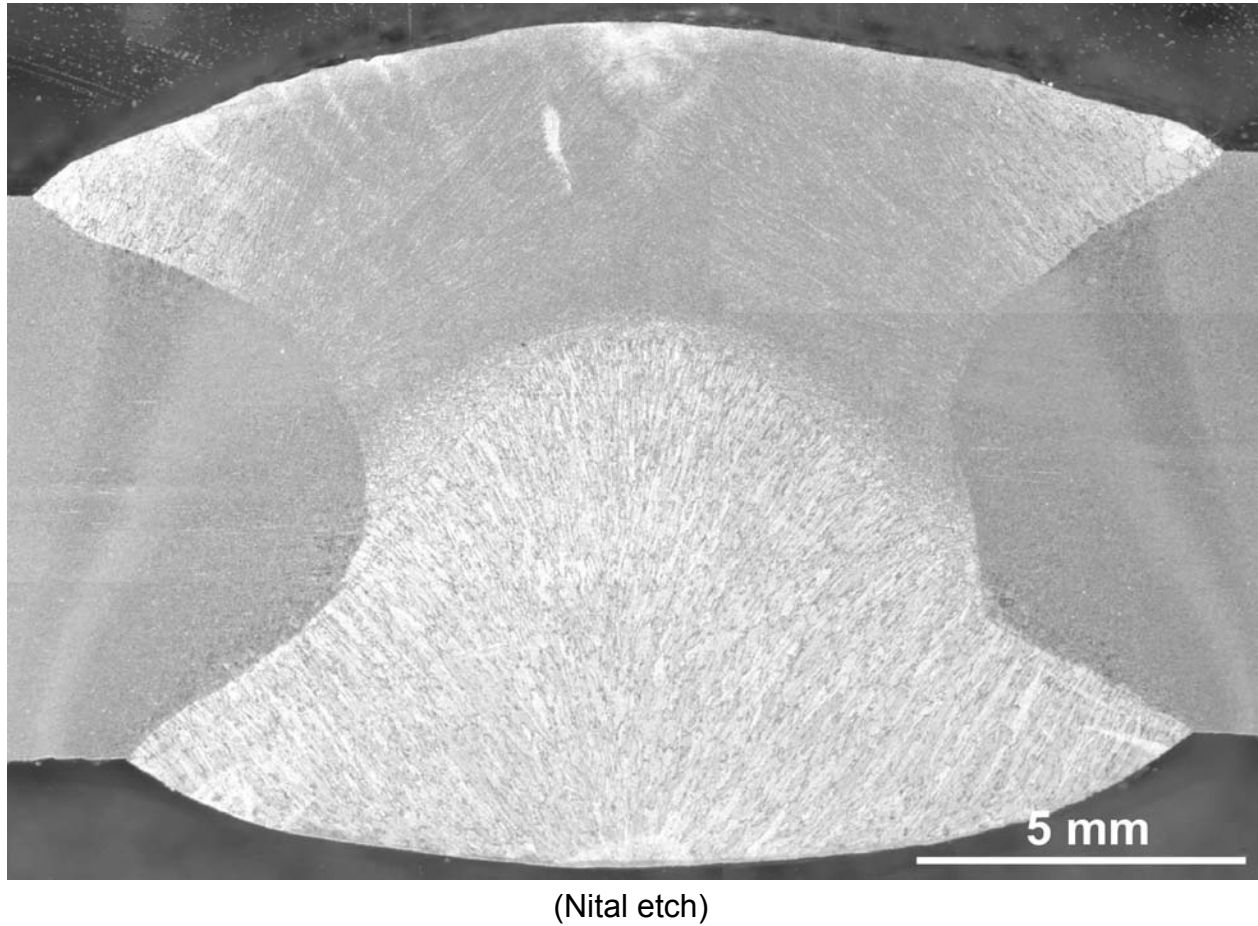


Figure 8. Photomicrograph of cross-section of weld.

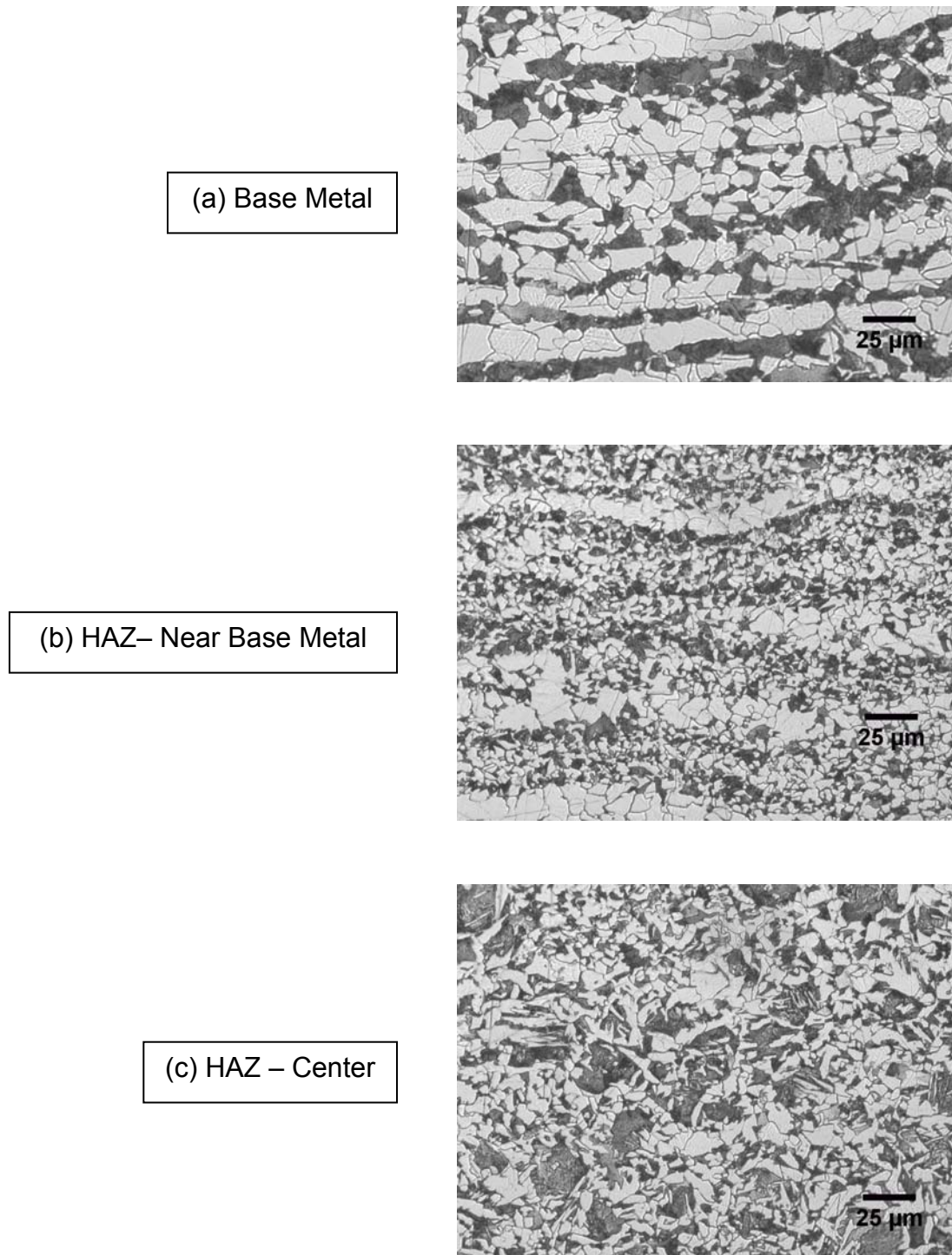
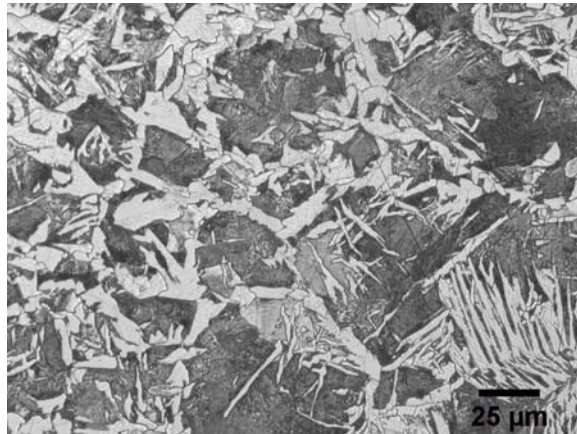
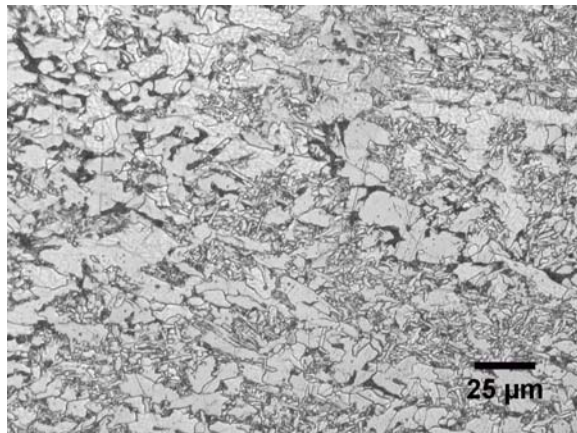


Figure 9. Optical photomicrographs showing grain structures visible at different locations in the plate (Nital etch).

(d) HAZ– Near Weld Metal



(e) Weld Metal – Near HAZ



(f) Weld Metal – Center
Weld Metal – Near HAZ

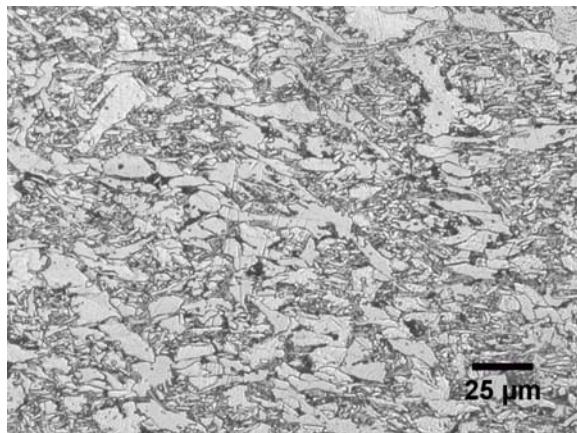


Figure 9. Optical photomicrographs showing grain structures
(continued) visible at different locations in the plate. (Nital etch)

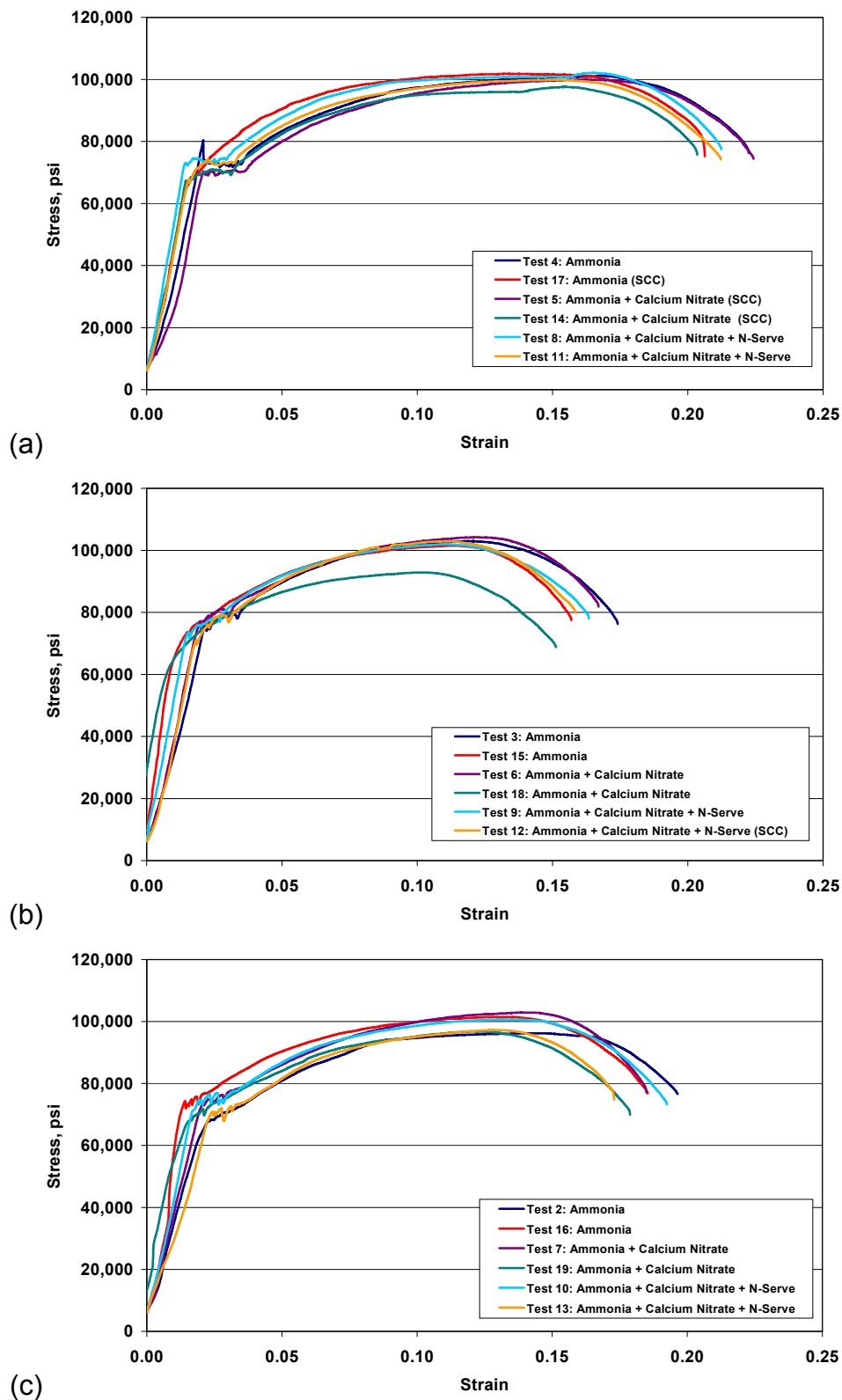
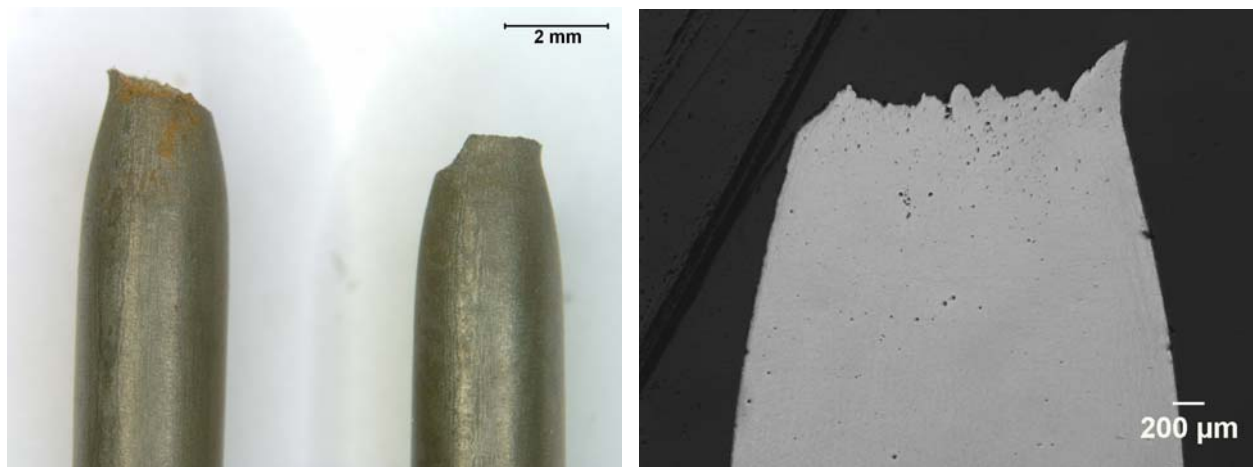
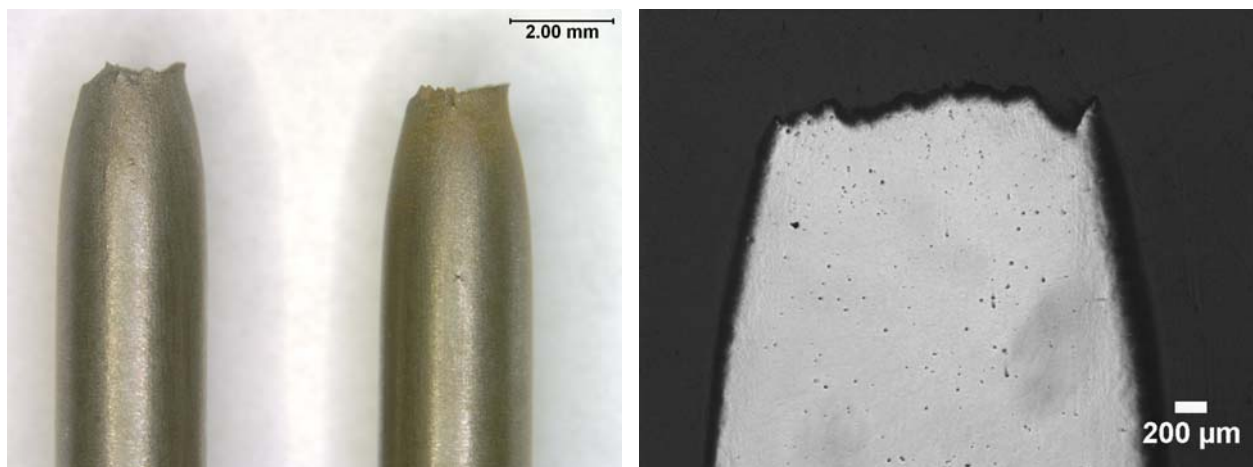


Figure 10. Stress-strain curves obtained in the different test environments for each of three materials: a) base metal, b) HAZ, c) weld fusion metal.



11(a) Test #4 – SA 455 Base Metal

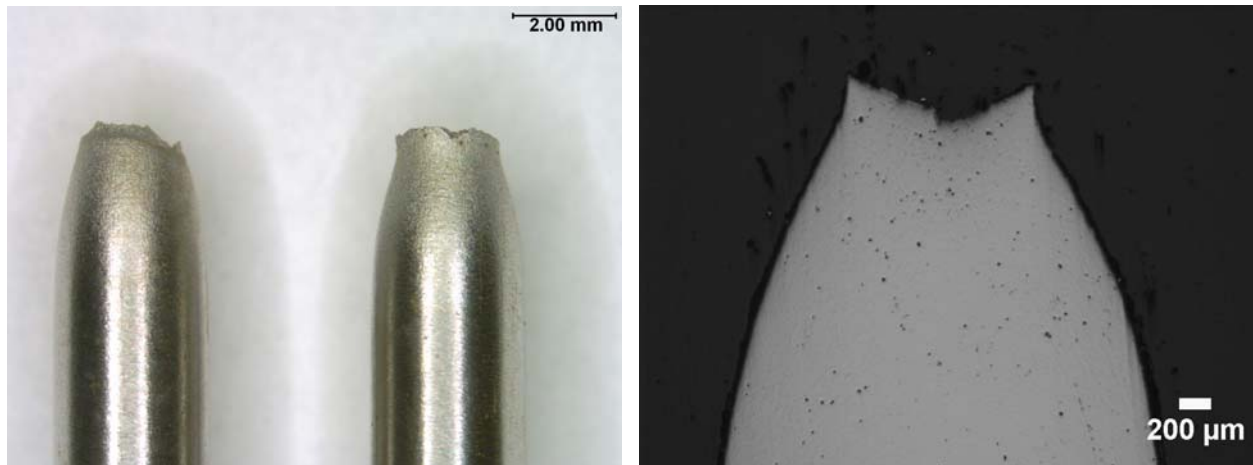


11(b) Test #17 – SA 455 Base Metal

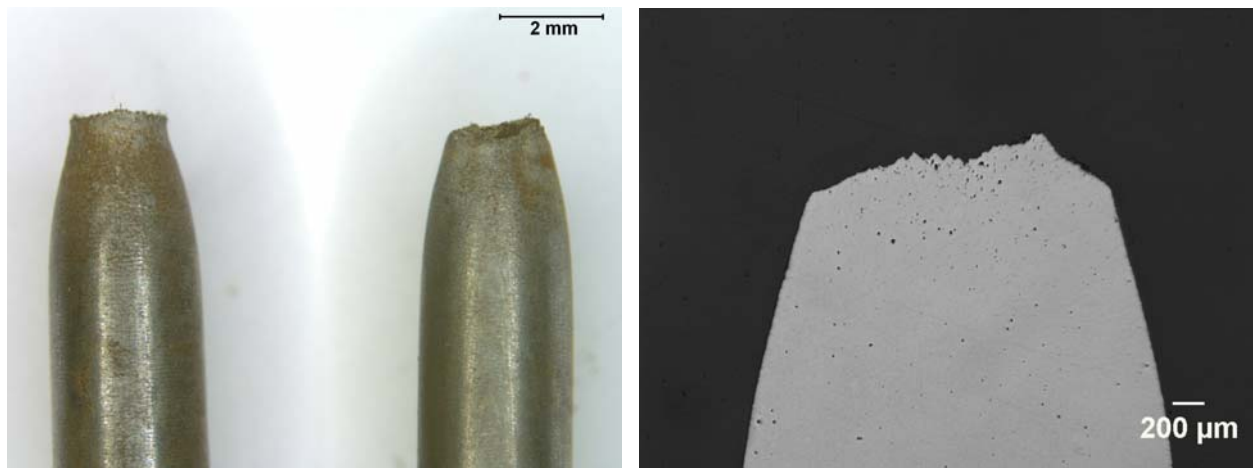


11(c) Test #2 – SA 455 HAZ (no PWHT)

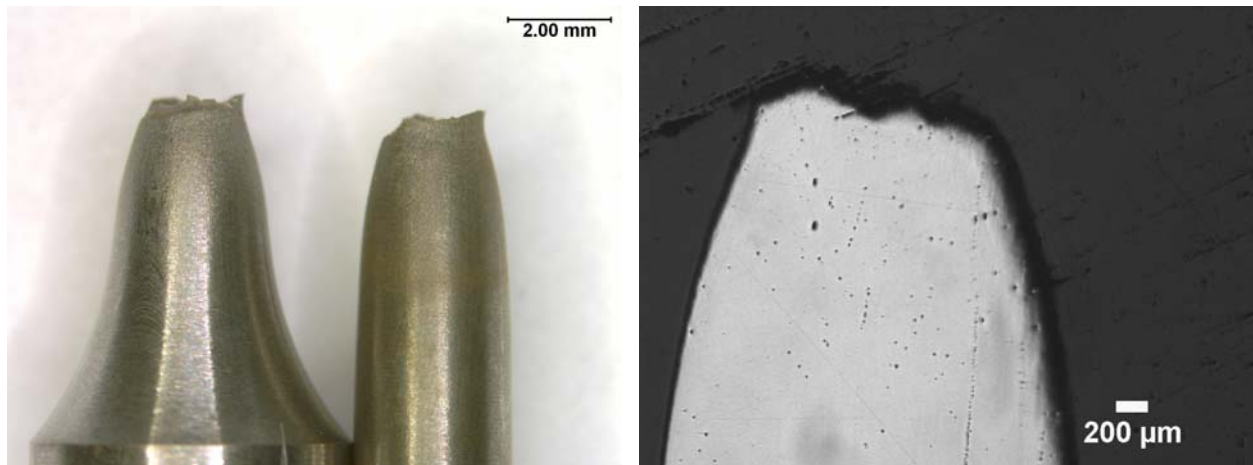
Figure 11. Tests in anhydrous ammonia – photographs of the fractured gage sections of the various specimens, and photomicrographs showing the respective fractured tips in longitudinal cross-sections, as-polished.



11(d) Test #15 – SA 455 HAZ (no PWHT)

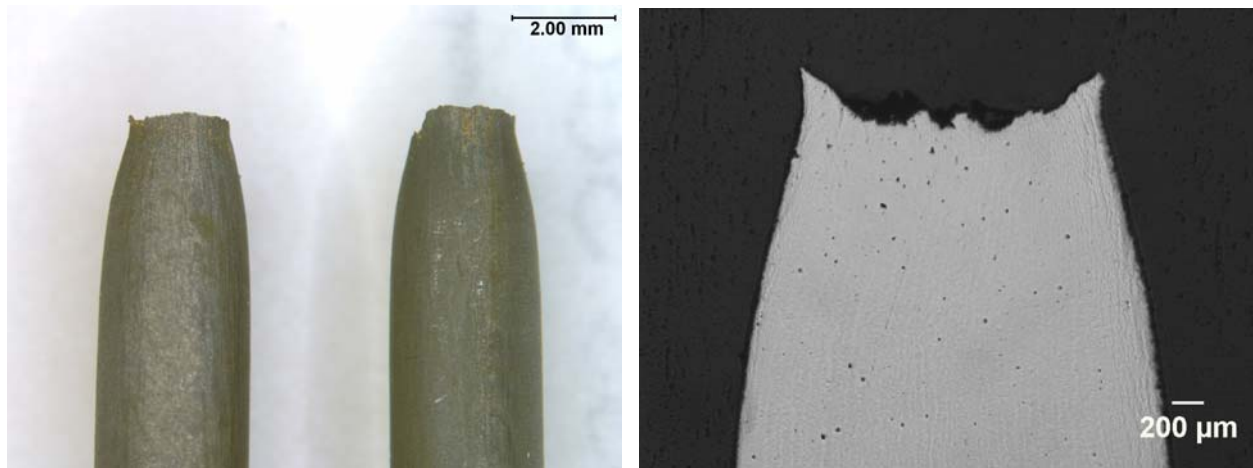


11(e) Test #3 – SA 455 Weld Metal (no PWHT)

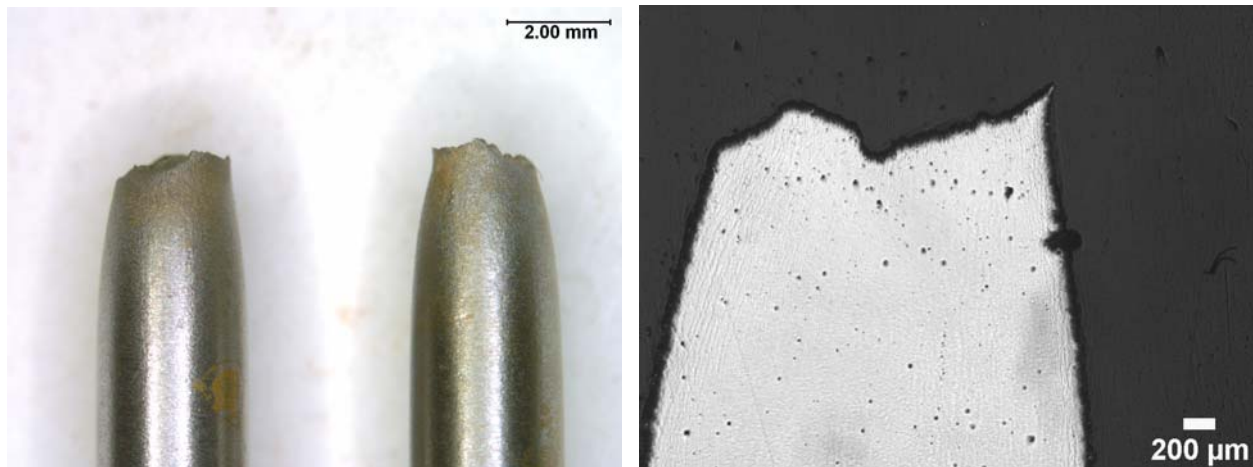


11(f) Test #16 – SA 455 Weld Metal (no PWHT)

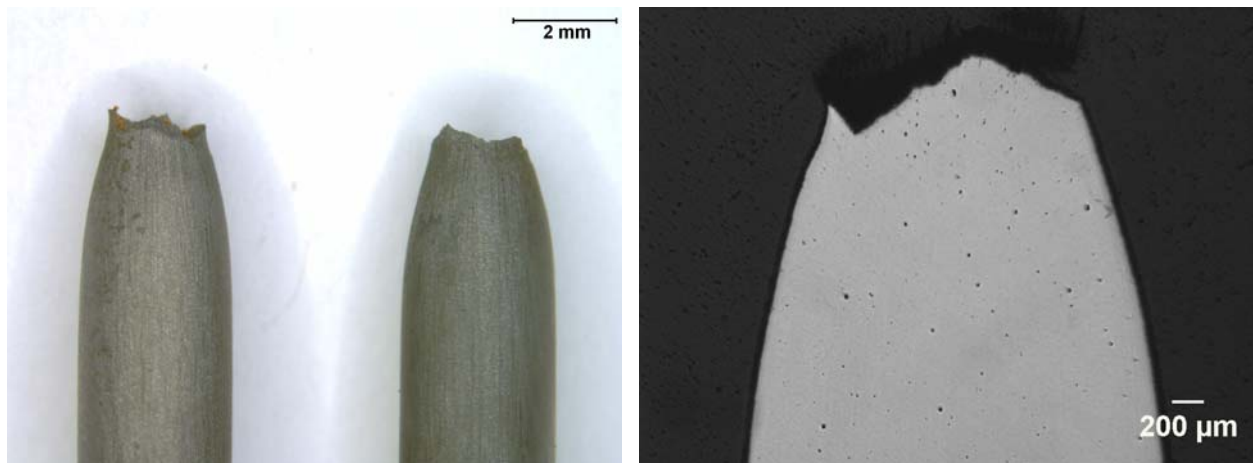
Figure 11. Tests in anhydrous ammonia – photographs of the fractured gage sections of the various specimens, and photomicrographs showing the respective fractured tips in longitudinal cross-sections, as-polished.
(continued)



12(a) Test #5 – SA 455 Base Metal

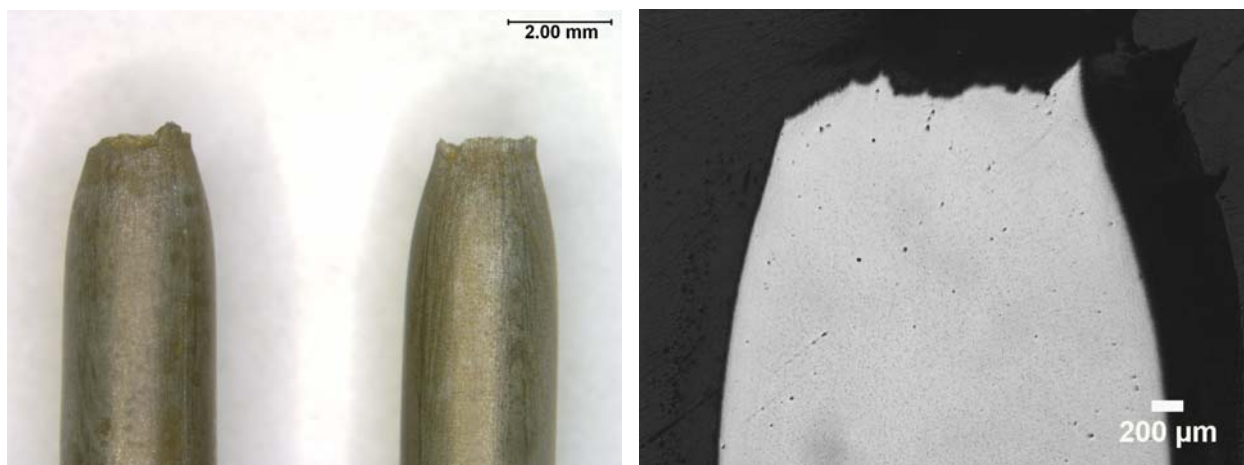


12(b) Test #14 – SA 455 Base Metal

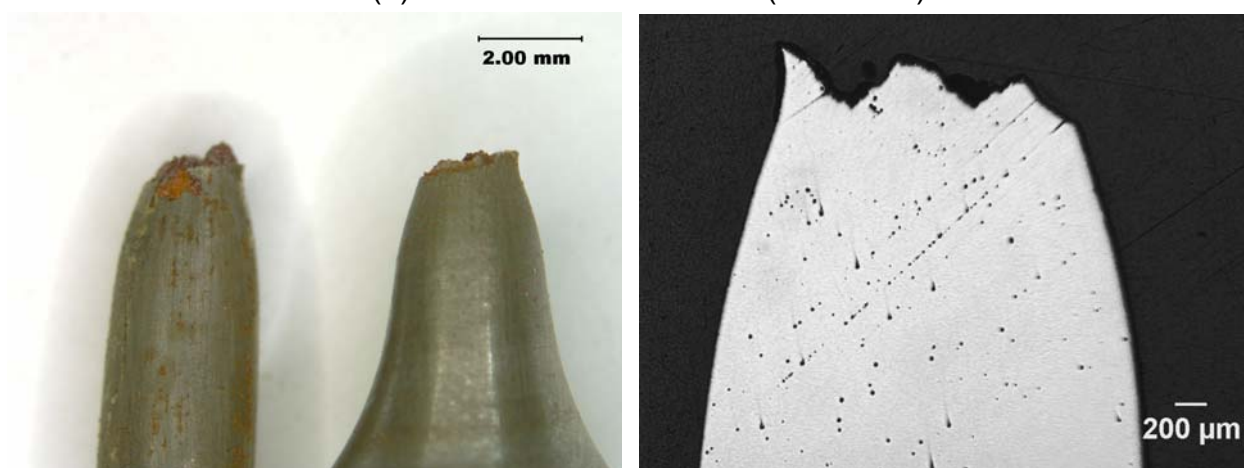


12(c) Test #6 – SA 455 HAZ (no PWHT)

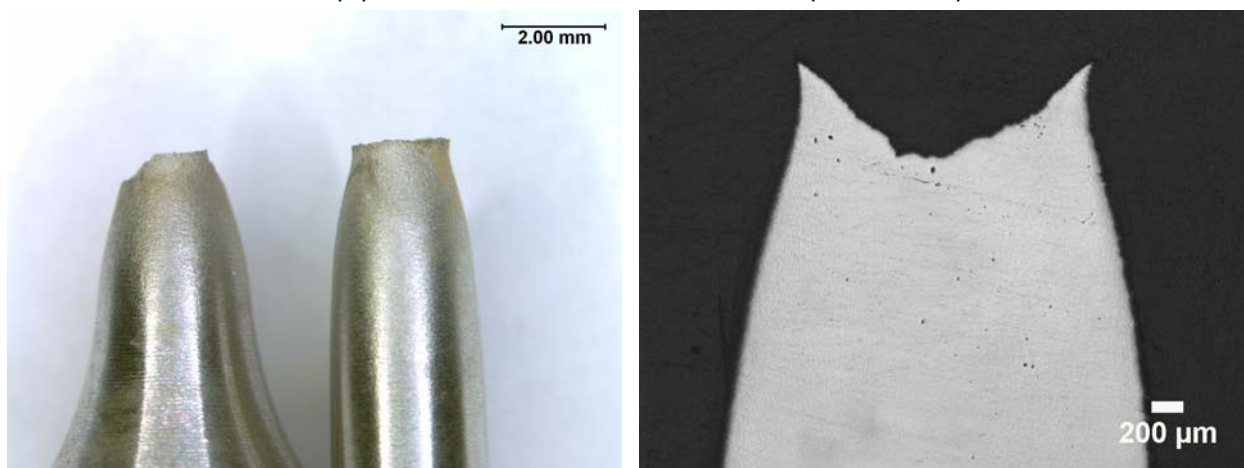
Figure 12. Tests in anhydrous ammonia with 2% $\text{Ca}(\text{NO}_3)_2$ – photographs of the fractured gage sections of the various specimens, and photomicrographs showing the respective fractured tips in longitudinal cross-sections, as-polished.



12(d) Test #18 – SA 455 HAZ (no PWHT)



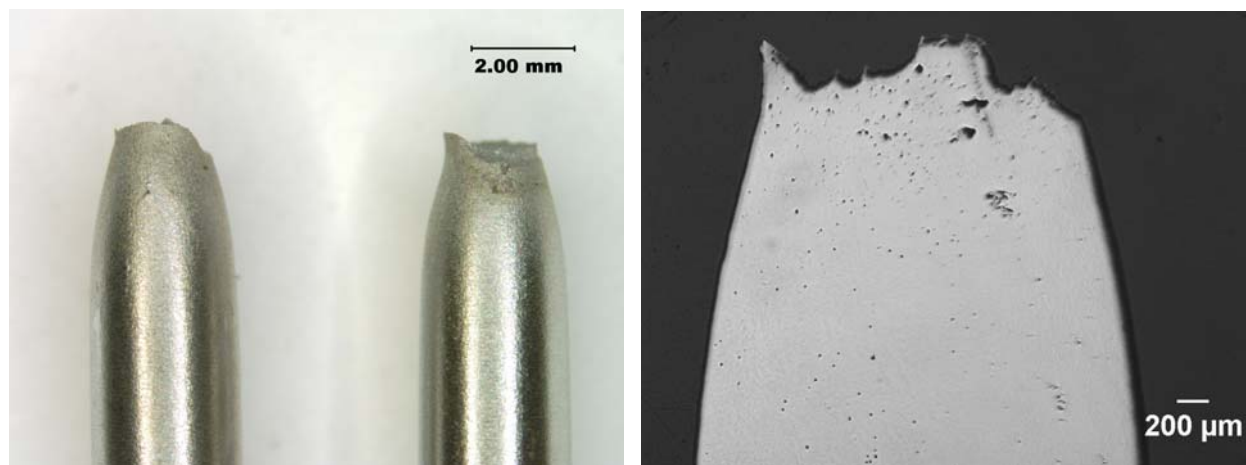
12(e) Test #7 – SA 455 Weld Metal (no PWHT)



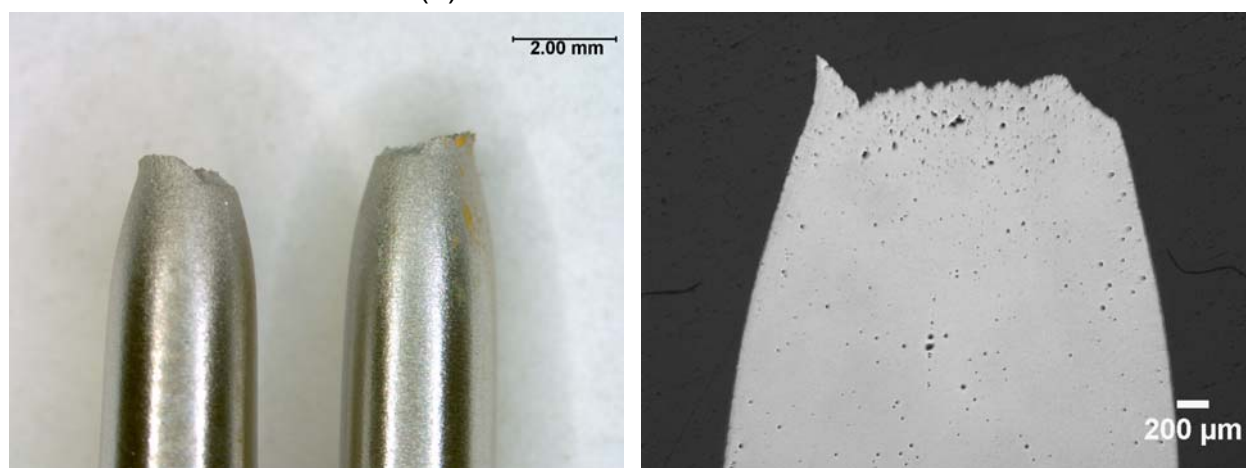
12(f) Test #19 – SA 455 Weld Metal (no PWHT)

Figure 12. Tests in anhydrous ammonia with 2% $\text{Ca}(\text{NO}_3)_2$ – photographs of the fractured gage sections of the various specimens, and photomicrographs showing the respective fractured tips in longitudinal cross-sections, as-polished.

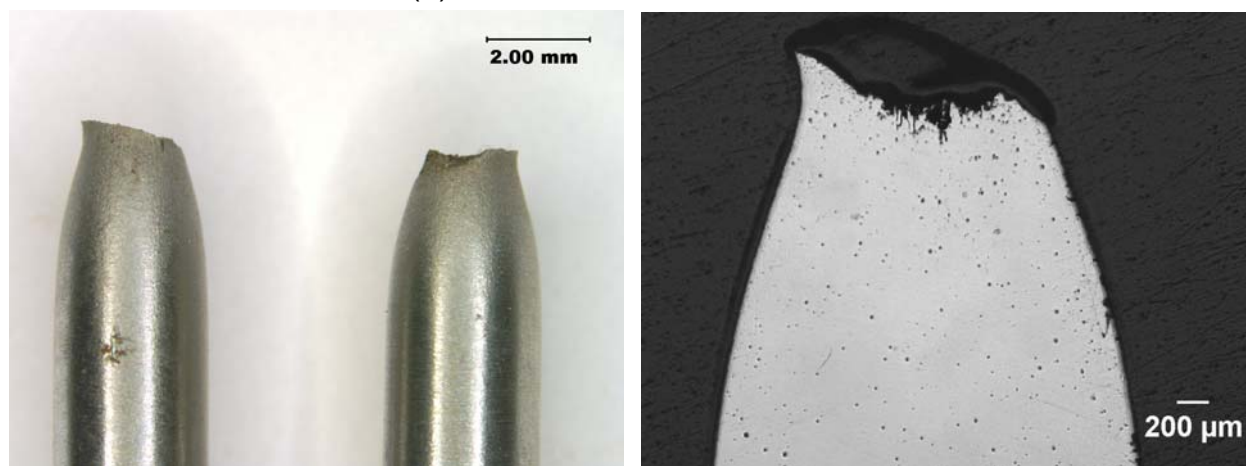
(continued)



13(a) Test #8 – SA 455 Base Metal

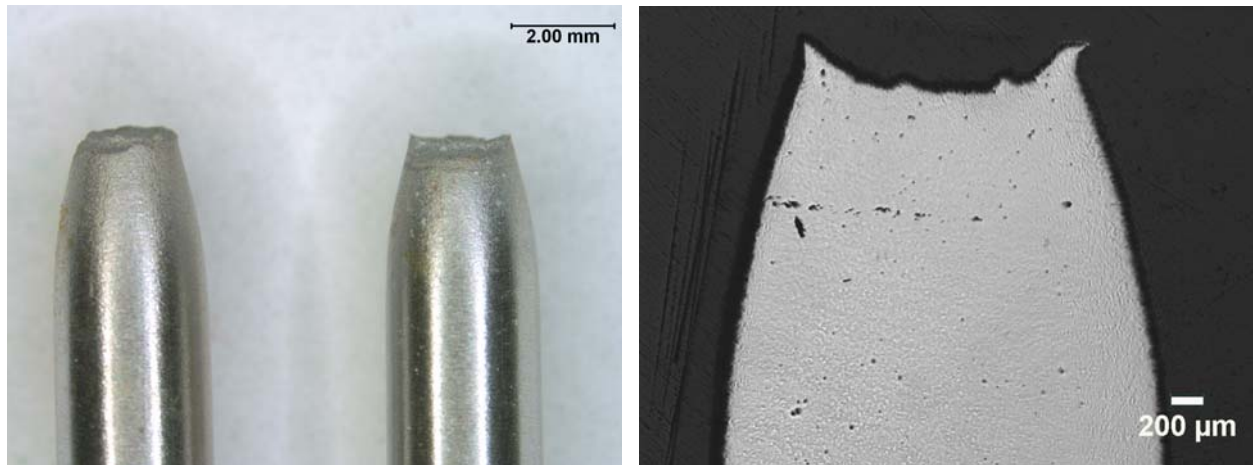


13(b) Test #11 – SA 455 Base Metal

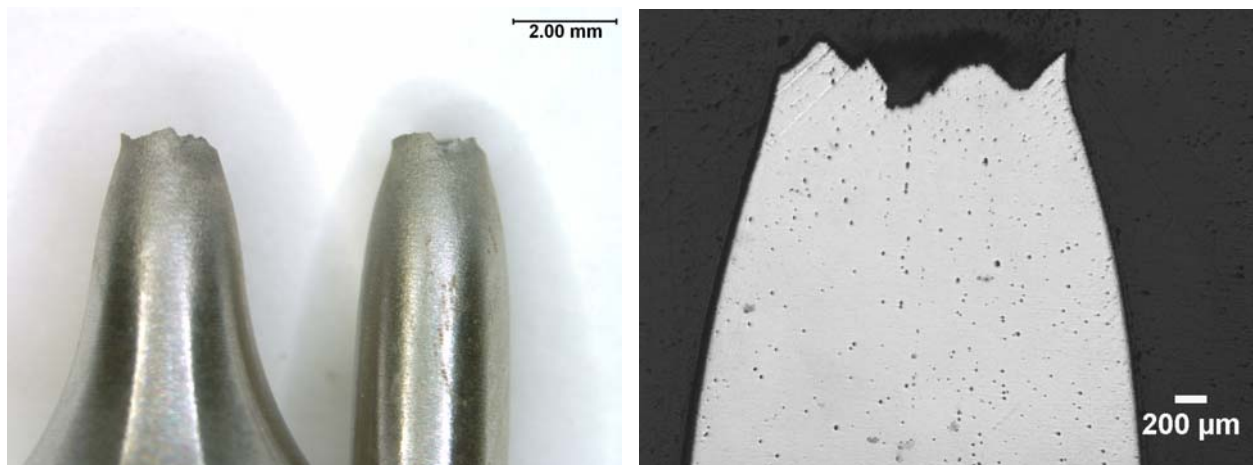


13(c) Test #9 – SA 455 HAZ (no PWHT)

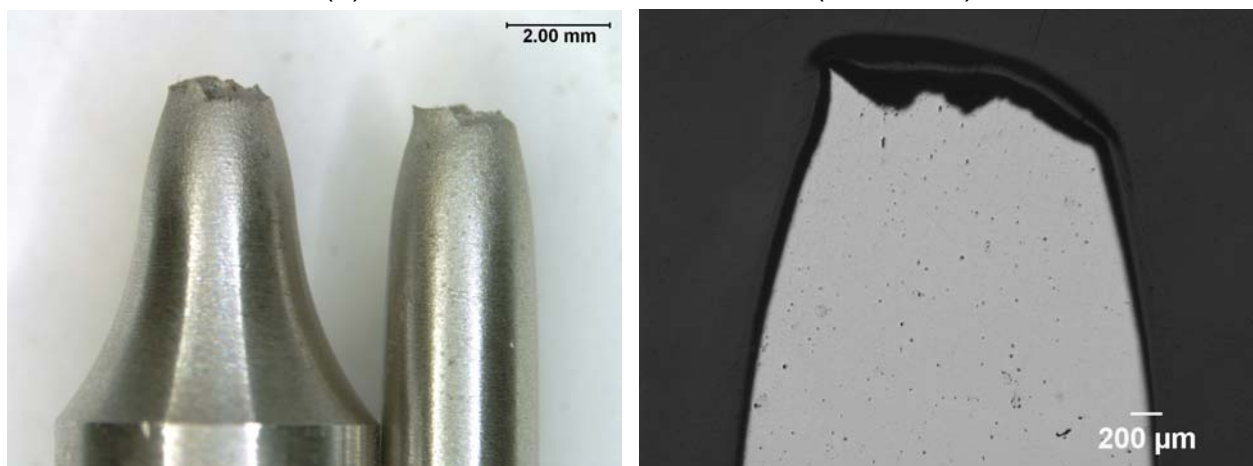
Figure 13. Tests in anhydrous ammonia with 2% $\text{Ca}(\text{NO}_3)_2$, and N-Serve® – photographs of the fractured gage sections of the various specimens, and photomicrographs showing the respective fractured tips in longitudinal cross-sections, as-polished.



13(d) Test #12 – SA 455 HAZ (no PWHT)

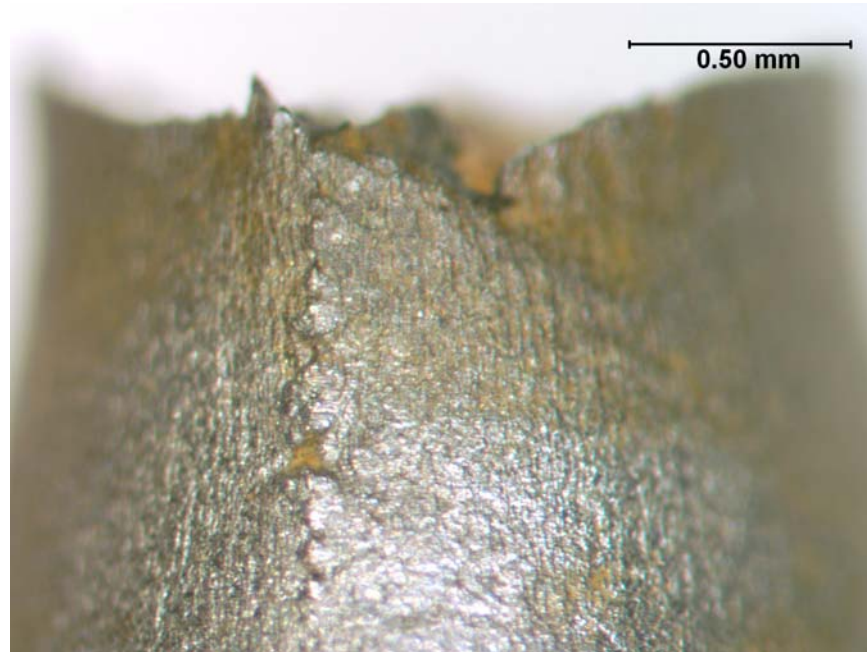


13(e) Test #10 – SA 455 Weld Metal (no PWHT)

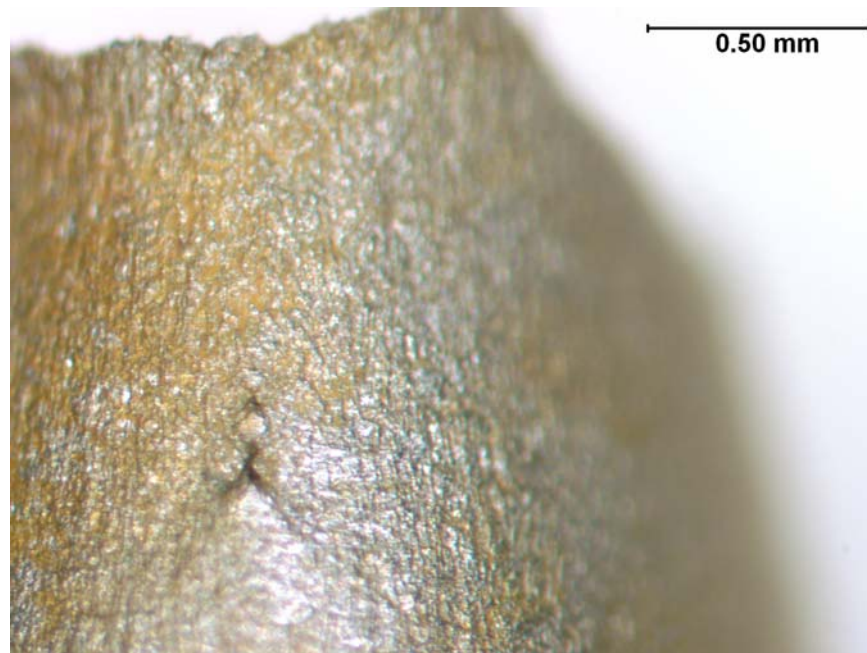


13(f) Test #13 – SA 455 Weld Metal (no PWHT)

Figure 13. Tests in anhydrous ammonia with 2% $\text{Ca}(\text{NO}_3)_2$, and N-Serve® –
(continued) photographs of the fractured gage sections of the various specimens, and
photomicrographs showing the respective fractured tips in longitudinal
cross-sections, as-polished.

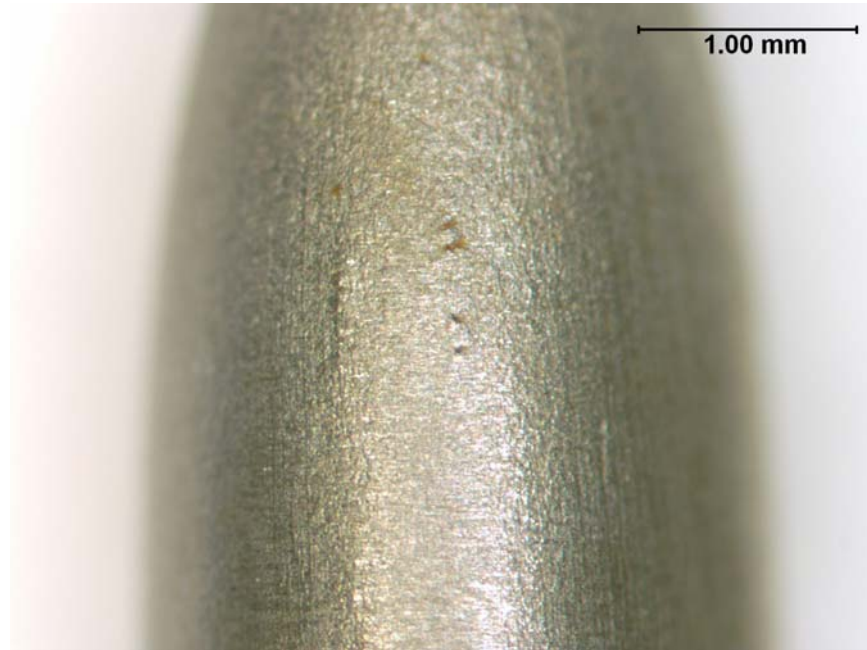


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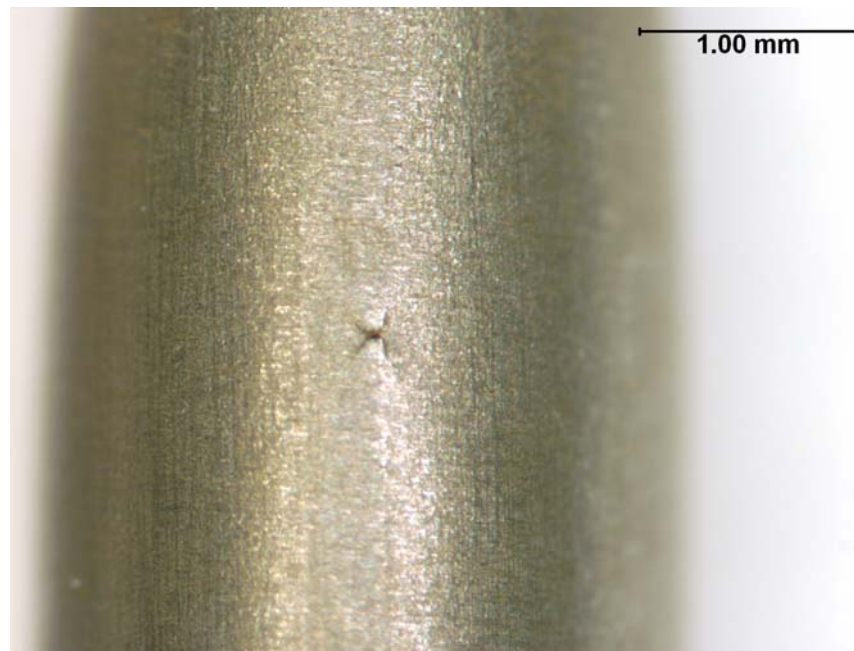


(b)

Figure 14. Stereo-photomicrographs of gage section near fracture surface of specimen from Test 14 showing small surface fissures.

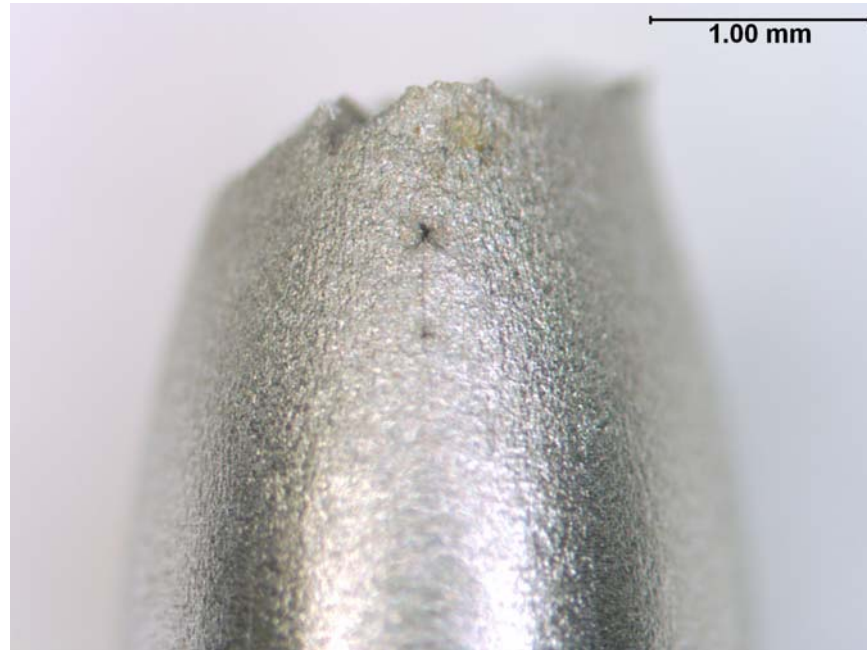


(a)



(b)

Figure 15. Stereo-photomicrographs of gage section near fracture surface of specimen from Test 17 showing small surface fissures.

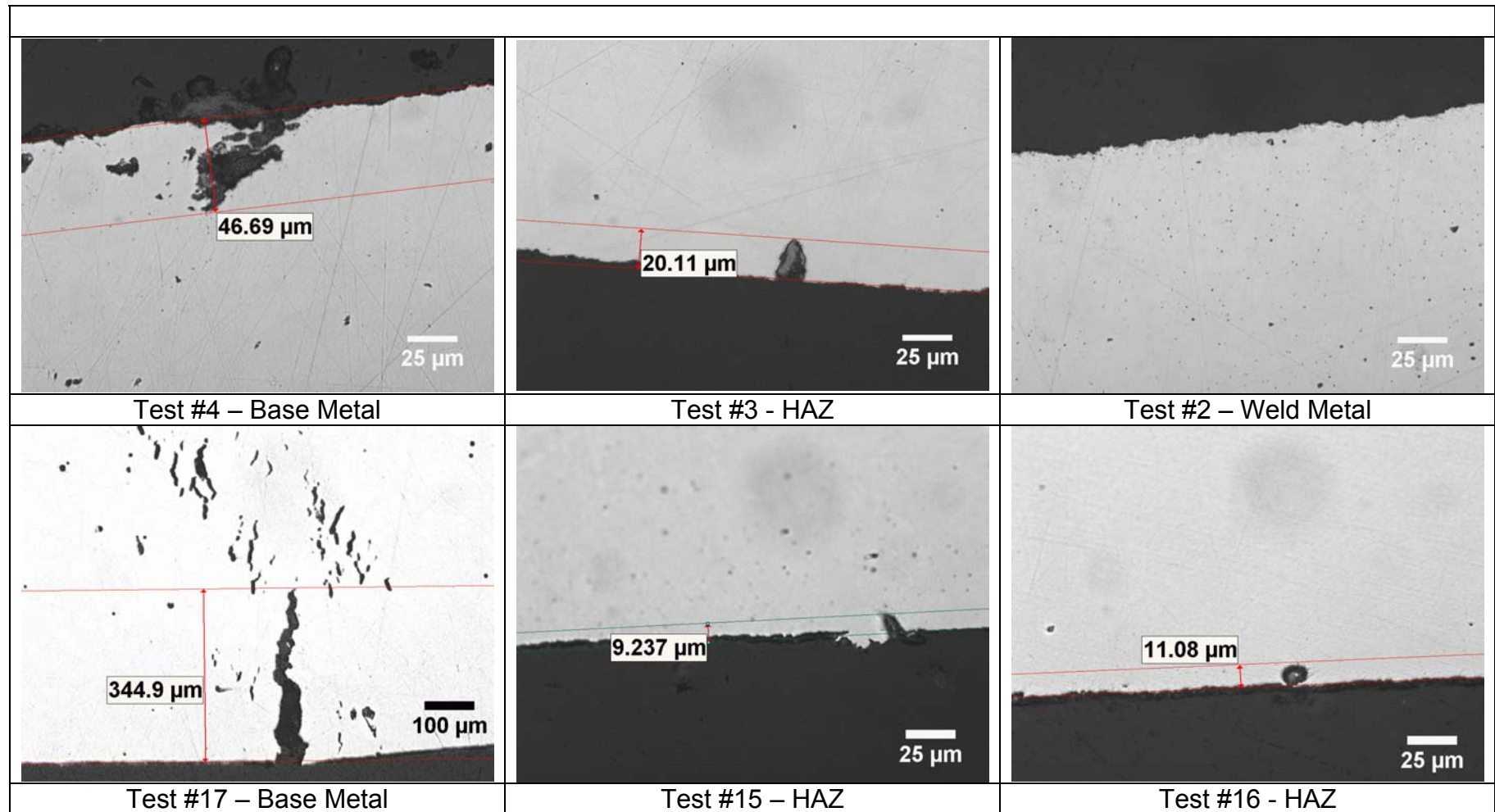


(a)



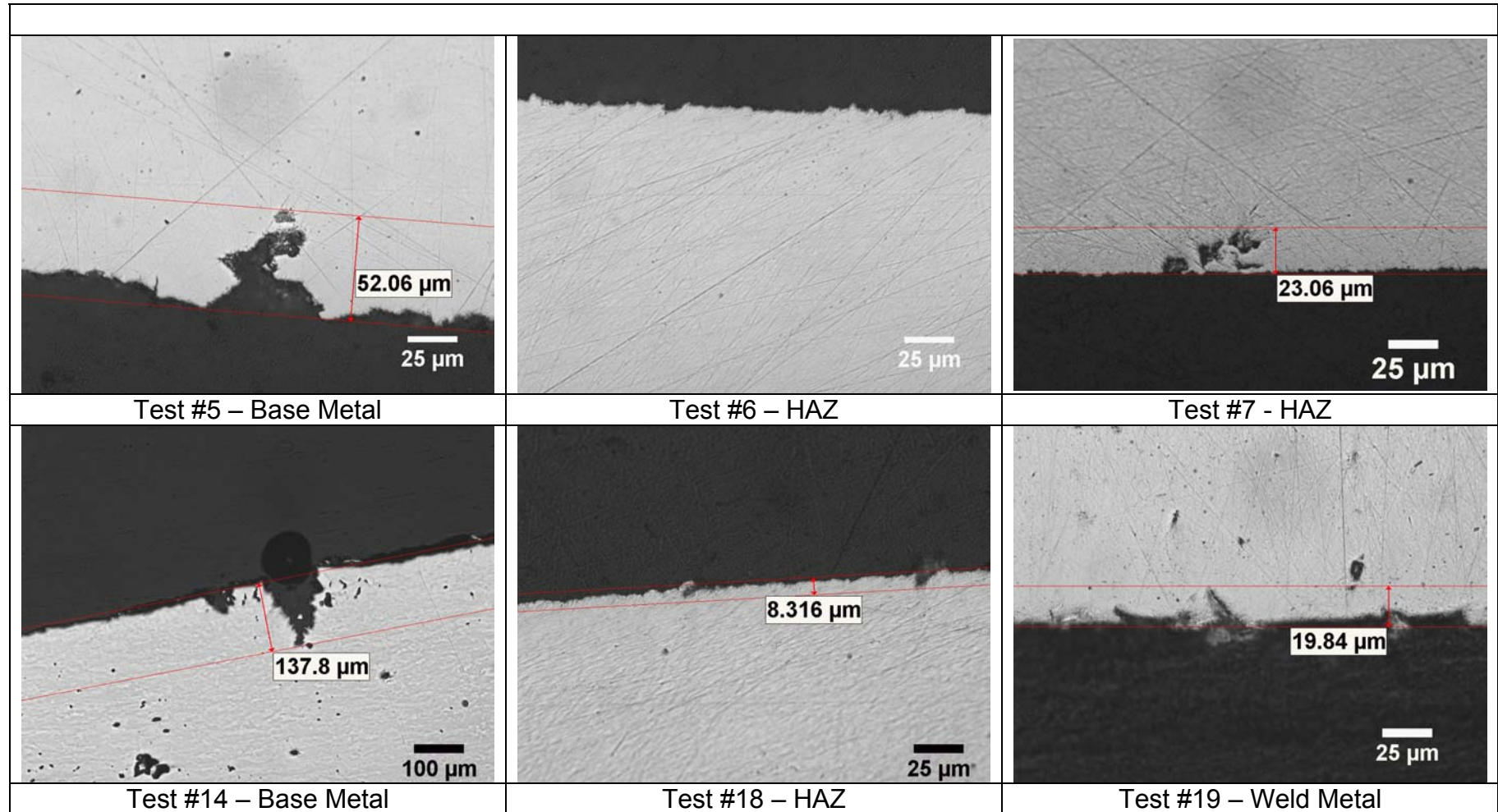
(b)

Figure 16. Stereo-photomicrographs of gage section near fracture surface of specimen from Test 12 showing small surface fissures.



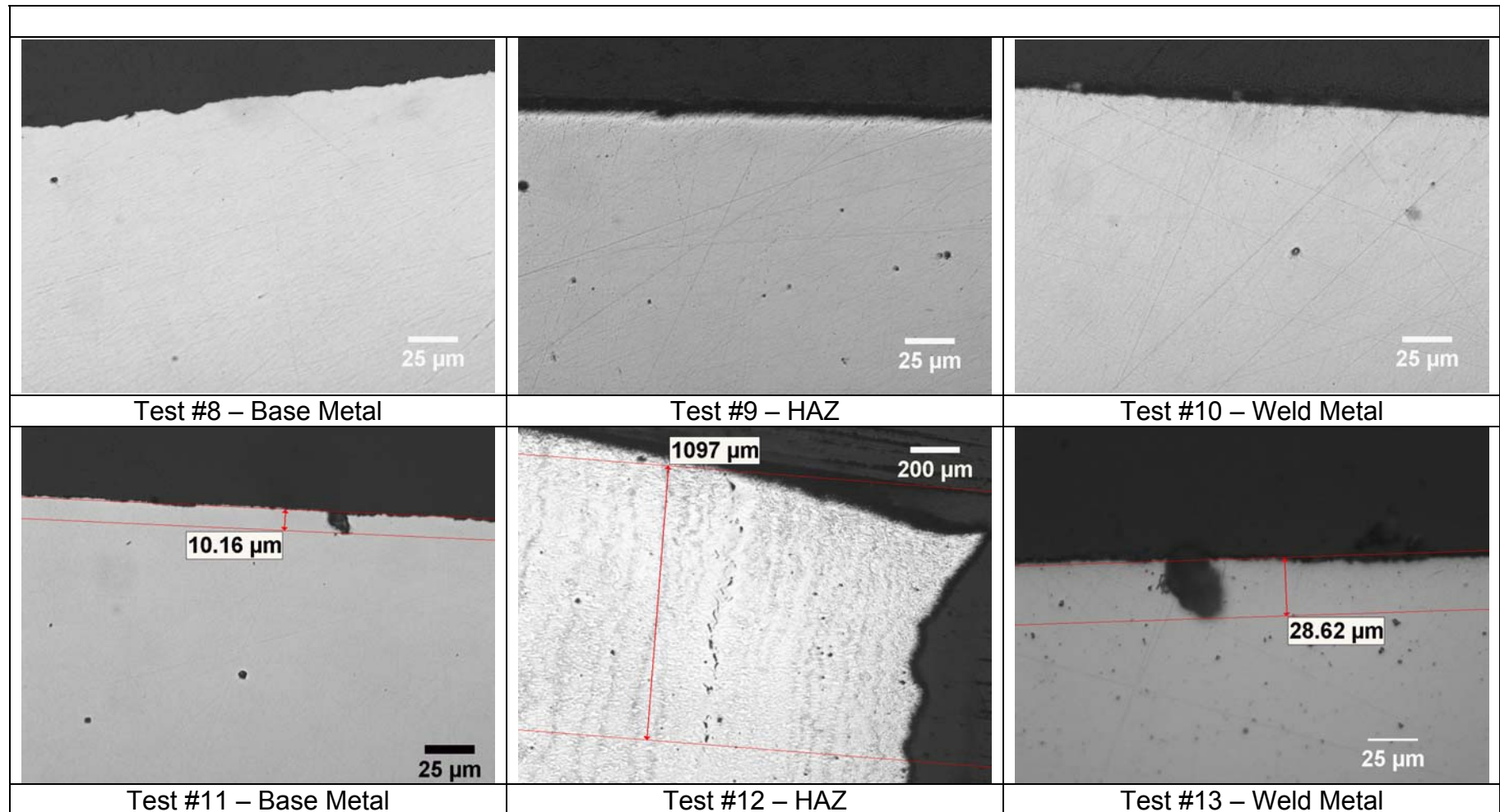
(a) Anhydrous Ammonia

Figure 17a. Photomicrographs showing the deepest surface fissures in gage sections of specimens tested in anhydrous ammonia.



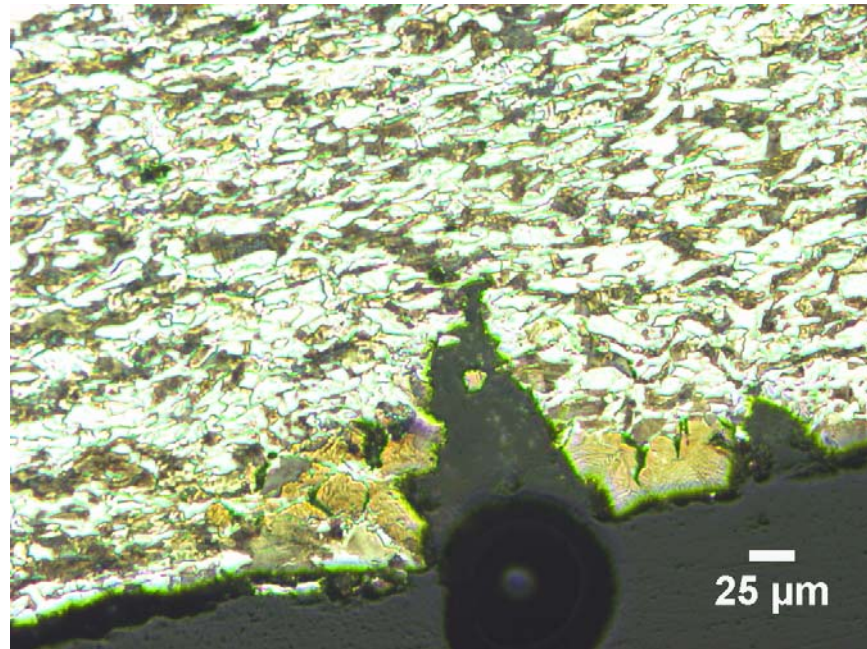
(b) Anhydrous Ammonia with 2% $\text{Ca}(\text{NO}_3)_2$

Figure 17b. Photomicrographs showing the deepest surface fissures in gage sections of specimens tested in anhydrous ammonia + 2% $\text{Ca}(\text{NO}_3)_2$.

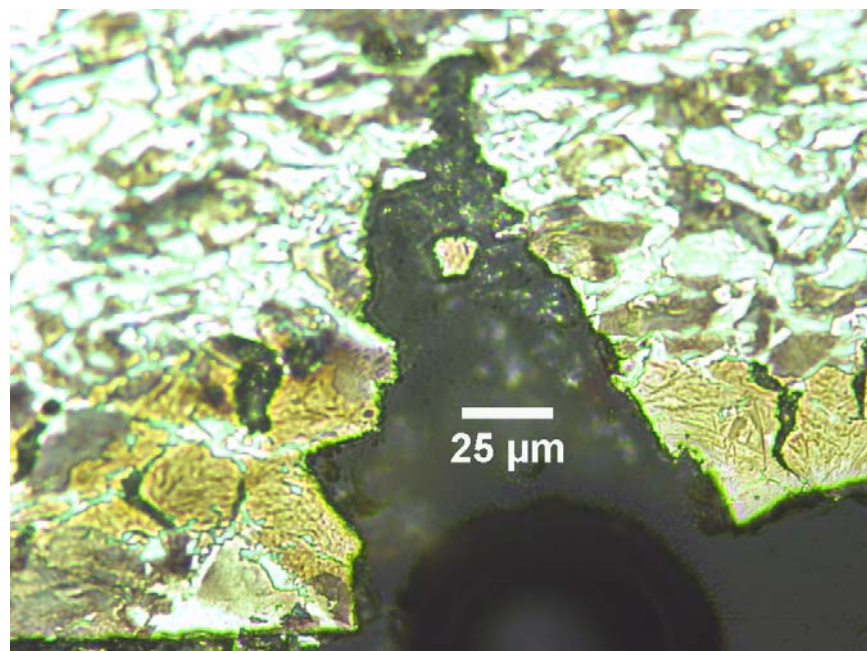


(c) Anhydrous Ammonia with 2% $\text{Ca}(\text{NO}_3)_2$ and N-Serve®

Figure 17c. Photomicrographs showing the deepest surface fissures in gage sections of specimens tested in anhydrous ammonia + 2% $\text{Ca}(\text{NO}_3)_2$ + N-Serve®.

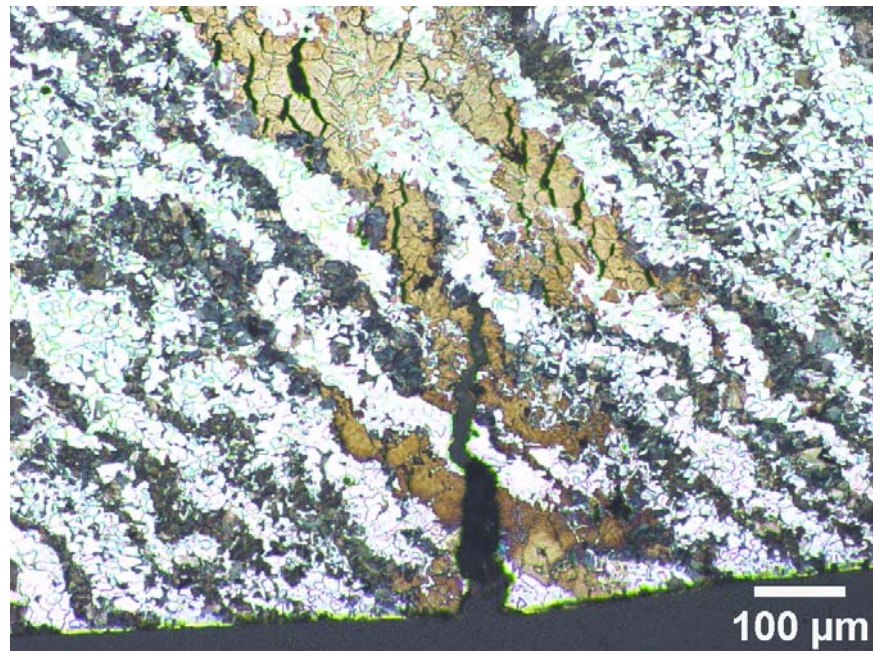


(a)

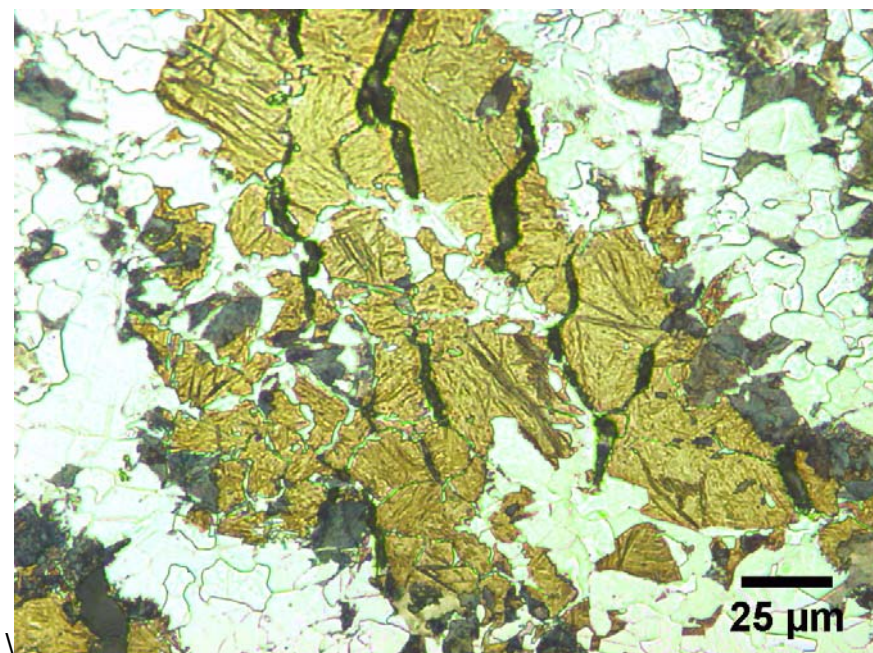


(b)

Figure 18. Photomicrographs of cross-section of gage section of specimen from Test 14 showing secondary cracks; Nital etched.

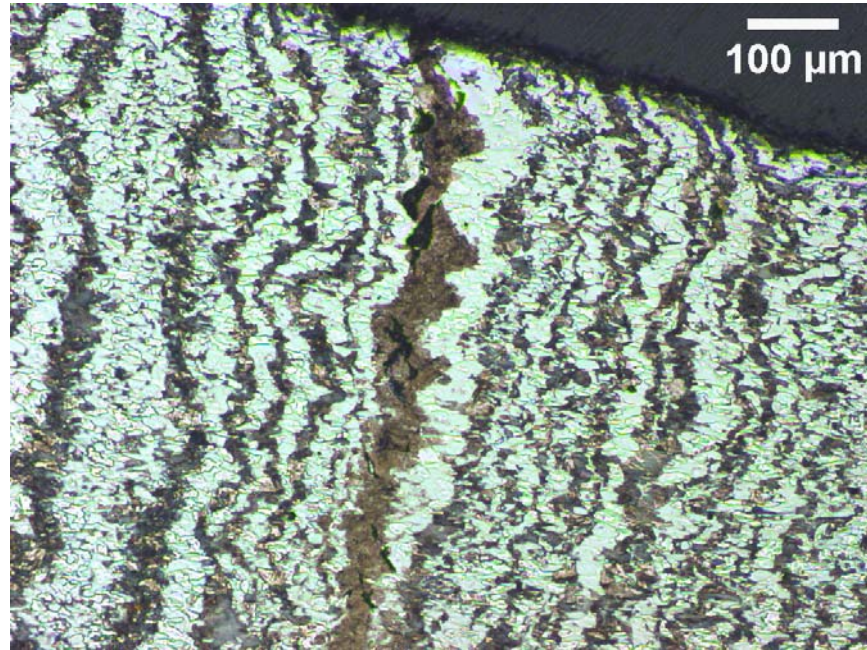


(a)

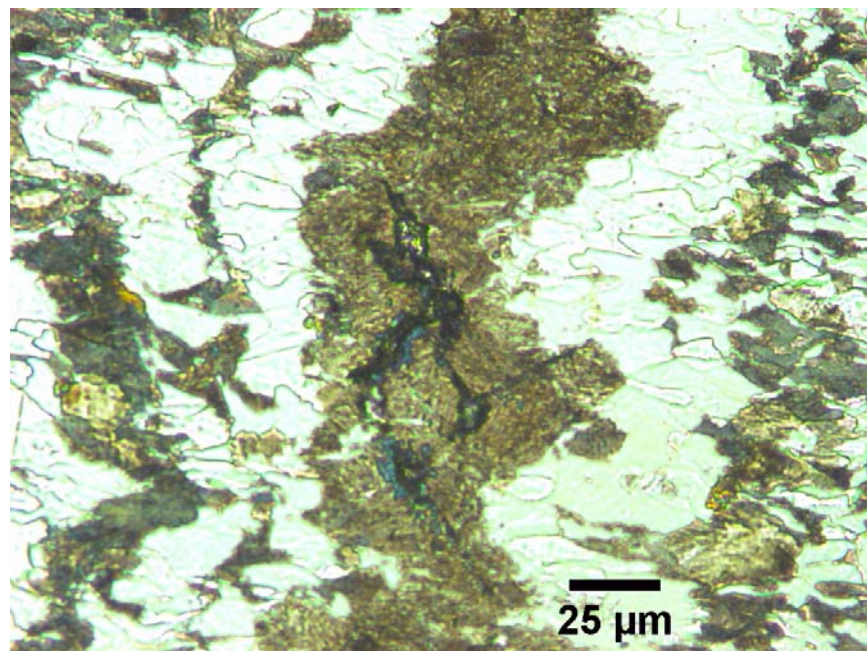


(b)

Figure 19. Photomicrographs of cross-section of gage section of specimen from Test 17 showing secondary cracks; Nital etched.



(a)



(b)

Figure 20. Photomicrographs of cross-section of gage section of specimen from Test 12 showing secondary cracks; Nital etched.

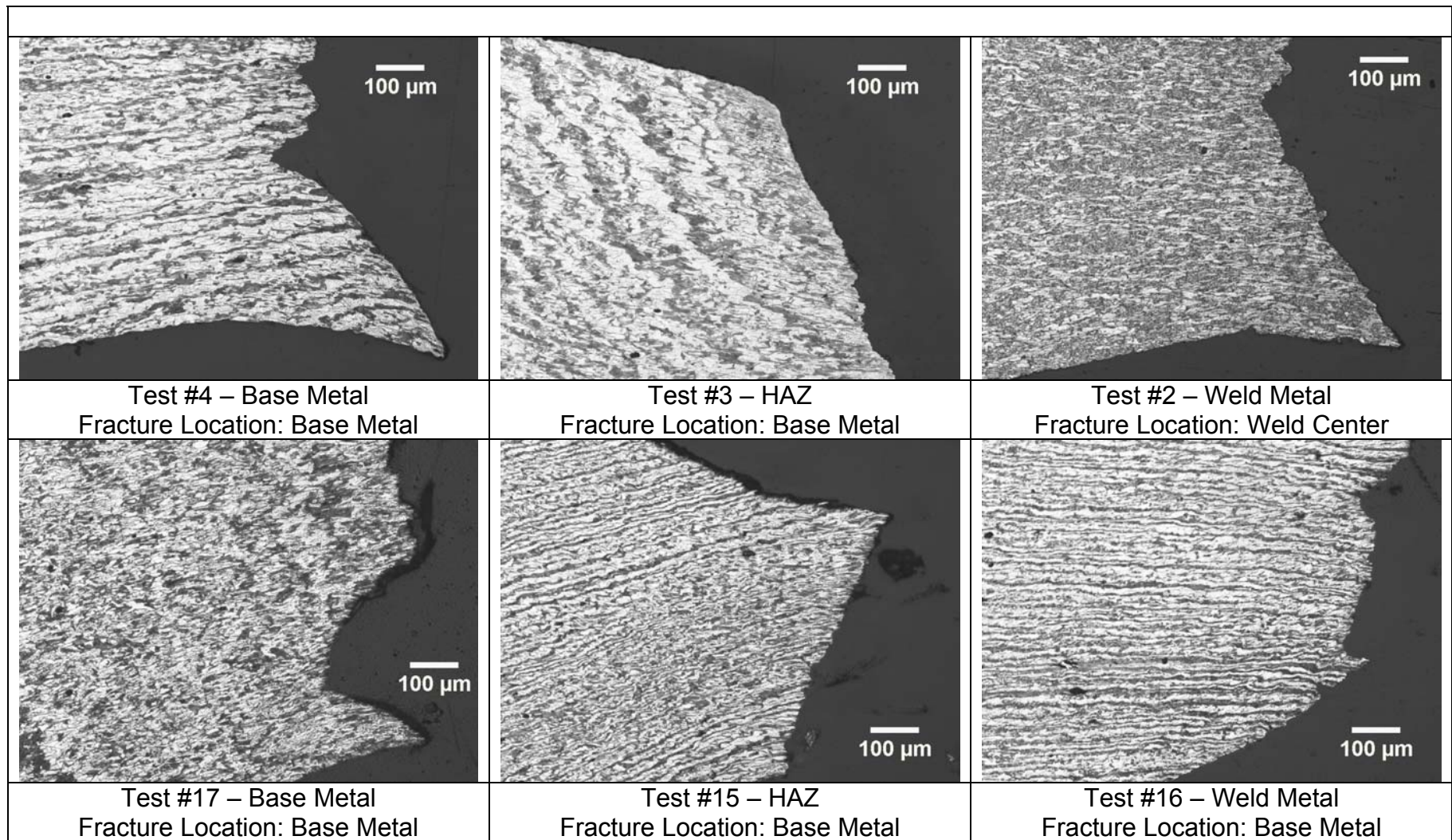
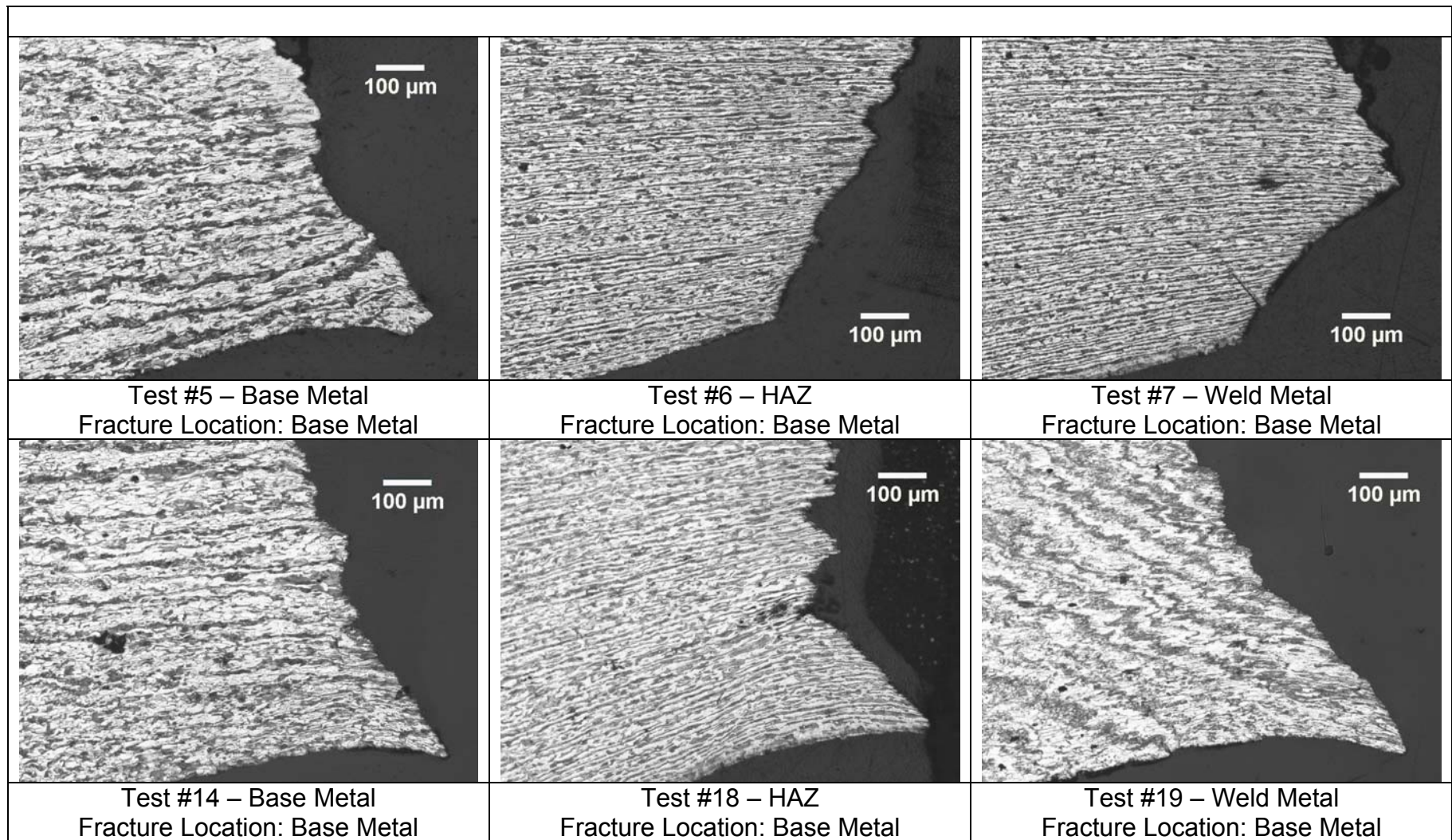
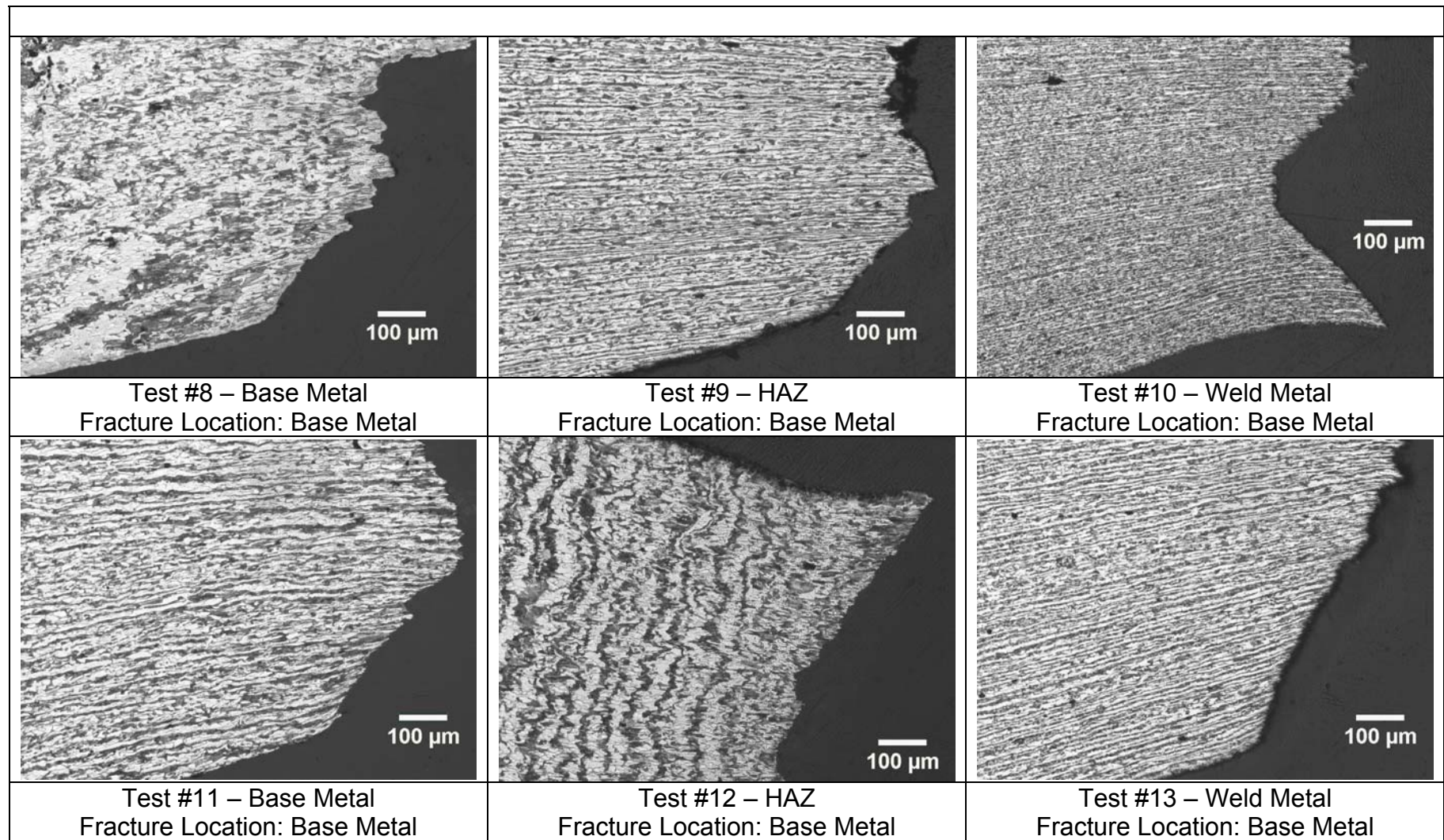
**(a) Anhydrous Ammonia**

Figure 21a. Photomicrographs showing the grain structure of specimens near the fracture location, in anhydrous ammonia; Nital etched.



(b) Anhydrous Ammonia with 2% $\text{Ca}(\text{NO}_3)_2$

Figure 21b. Photomicrographs showing the grain structure of specimens near the fracture location, in anhydrous ammonia + 2% $\text{Ca}(\text{NO}_3)_2$; Nital etched.



(c) Anhydrous Ammonia with 2% $\text{Ca}(\text{NO}_3)_2$ and N-Serve®

Figure 21c. Photomicrographs showing the grain structure of specimens near the fracture location, in anhydrous ammonia + 2% $\text{Ca}(\text{NO}_3)_2$ + N-Serve®; Nital etched.

APPENDIX A

PROCEDURE FOR SCC TESTS IN ANHYDROUS AMMONIA

1.0 SSRT System Setup

- 1.1 Use System #3 located in fume hood 1W3
- 1.2 Check that fume hood is working
- 1.3 Fill out the applicable boxes of the Test Information Form
- 1.4 Check for current load cell, LVDT, and motor calibration
- 1.5 Initiate data acquisition system & check that load cell and LVDT are properly connected
- 1.6 Set RPM at 691 for 1×10^{-6} in/in/sec strain rate
- 1.7 Reposition the lower jackscrew for a minimum of 1 in. down travel
- 1.8 Verify that the LVDT has stroke length of at least 0.5 in.

2.0 Specimen Preparation

- 2.1 Select the specified specimen from the stock
- 2.2 Measure and record the distance between the end points
- 2.3 Use gloves to handle the specimen from here on
- 2.4 Degrease the specimen with acetone
- 2.5 Place a rusted spring around the specimen
- 2.6 Screw the specimen with spring into pull rods

3.0 Cell assembly

- 3.1 Check that the test cell is equipped with various fittings and valves per the drawing
- 3.2 (Note: only stainless steel fittings, valves and gages are allowed)
- 3.4 Carefully insert the pull rods through the Conax glands into the cell
- 3.5 Position the specimen in the center of the cell
- 3.6 Tighten the gland nuts on both ends finger-tight + 1-1/4 turn
- 3.7 Place the assembled cell on the wooden support platform in the load frame
- 3.8 Connect the lower pull rod to jackscrew with a clevis joint & ceramic coated pin
- 3.9 Connect upper pull rod to top jackscrew using a universal joint & ceramic coated pin
- 3.10 Verify electrical isolation of the pull rods from the SSR system using a VOM
- 3.11 Remove slack from the load train by carefully turning wheel-nut on the SSR frame

4.0 Pressure Testing Cell

- 4.1 Set regulator at 135 psig on argon cylinder
- 4.2 Close top cell valve
- 4.3 Open bottom cell valve
- 4.4 Connect hose from argon cylinder to the bottom cell valve
- 4.5 Open needle valve gently to let in argon from tank into the cell
- 4.6 Close bottom cell valve at 135 psig cell pressure
- 4.7 Close needle valve at argon cylinder
- 4.8 Check pressure after one hour, if no drop -> then cell is ready for next steps
- 4.9 Check and correct for leaks if pressure drops
- 4.10 Repeat pressure test if necessary

5.0 Evacuating Cell

- 5.1 Release the pressure from the cell
- 5.2 Purge cell for 5-10 minutes with Argon
- 5.3 Purge line to ammonia by loosening nut
- 5.4 Leave 5 psi in the cell, shut all valves to the cell and tighten nut on ammonia line.

6.0 Filling Cell with Ammonia

- 6.1 Connect top cell valve to discharge hose immersed in water filled carboy - Figure 1
- 6.2 Place hot water bag on ammonia cylinder and wait 10-15 minutes
- 6.3 Open the ammonia tank valve slowly
- 6.4 Fill cell to pre-marked height by opening/closing top cell valve to relieve head pressure
- 6.5 Close ammonia tank valve
- 6.6 Connect hose from PRV to water filled carboy
- 6.7 Cell is ready for the SSR test

7.0 Running the SSR Test

- 7.1 Adjust tension on the pull rods to 50 lbs, do not overload
- 7.2 Check the following:
 - 7.2.1 -Liquid level in cell
 - 7.2.2 -Cell pressure
 - 7.2.3 -LVDT reading
 - 7.2.4 -Preload reading
- 7.3 Close the fume hood doors
- 7.4 Turn on the motor and record time/date

8.0 Terminating the Test

- 8.1 Turn off motor on specimen rupture
- 8.2 Save data as necessary
- 8.3 Crack open top cell valve and discharge ammonia into water
- 8.4 Crack open bottom cell valve to empty ammonia feed line into cell and then water
- 8.5 After 2-4 hours, close the two valves and disconnect the cell from the discharge lines
- 8.6 Remove the cell from the test frame and place in a running hood.
- 8.7 Open valve to vent remaining pressure; cell will still have some ammonia inside
- 8.8 Disengage the pull rods and remove the cell from the SSR frame
- 8.9 Loosen the gland nuts and slide out the pull rods and fractured specimen
- 8.10 Retrieve the specimen from the pull rods, clean in DI H₂O, dry and store in dessicator

9.0 Specimen Evaluation

- 9.1 Visually examine the fracture pieces under a stereomicroscope for secondary cracks
- 9.2 Photograph the fractured pieces and show the secondary cracks if present
- 9.3 Measure/record the length between the reference points by joining the fractured pieces
- 9.4 Measure and record the gage diameter at one fracture tip
- 9.5 Fill the applicable boxes of the Test Information Form

10.0 Metallographic Examination

- 10.1 Cut off the gage section from one of the fractured pieces
- 10.2 Metallographically mount the above gage section axially
- 10.3 Grind and polish the gage section to its mid diameter while checking for cracks
- 10.4 Take photomicrographs of the cracks if present, & stamp micron markers
- 10.5 Etch with Nital and retake photomicrographs of the cracks & stamp micron markers
- 10.6 Complete the test information form with the metallographic data

11.0 Waste Disposal

- 11.1 Neutralize the aqueous ammonia solution to pH 12 with 10% muriatic acid
- 11.2 Drain the pH 12 solution into sink with plenty of running water
- 11.3 Record waste disposal in the lab book